TABLE OF CONTENTS

TABLES

ACRONYMS

ALARA	as low as reasonably achievable
ASTM	American Society for Testing and Materials
CCV	continuing calibration verification
CERCLA	*Comprehensive Environmental Response, Compensation, and Liability Act of 1980*
COC	contaminants of concern
DOE	U.S. Department of Energy
DQA	data quality assessment
DQO	data quality objective
EPA	U.S. Environmental Protection Agency
FSS	final status survey
GC	gas chromatograph
HASQARD	*Hanford Analytical Services Quality Assurance Requirements Document*
HSA	historical site assessment
ICP	inductively coupled plasma
IDL	instrument detection limit
LCS	laboratory control sample
LFL	lower flammability limit
MDA	minimum detectable activity
MDC	minimum detectable concentration
MDL	minimum detection level
MS	mass spectrometer
PCB	polychlorinated biphenyl
QA	quality assurance
QC	quality control
RCRA	*Resource Conservation and Recovery Act of 1976*
SAP	Sampling and Analysis Plan
TSD	treatment, storage, and disposal

VOLUME 3:

1.0   INTRODUCTION

The Hanford Site clean-up mission requires the flexibility to select semi-quantitative or quantitative methods for analysis of environmental samples to meet the project objectives. This volume sets forth the quality assurance (QA) and quality control (QC) requirements that govern analytical work usually performed using semi-quantitative approaches. Volume 4 establishes requirements for analyses usually conducted using quantitative methods.

As a matter of convention in this document, analysis performed directly on the sample or object as they exist in the field is referred to as *in situ analysis.* Analysis performed on samples collected in the field and analyzed in a field laboratory located at or near the sample collection point(s) is referred to as *field analysis.* Samples collected in the field and transported to fixed laboratory facilities located some distance from the sampling location are submitted for *laboratory analysis*. It must be emphasized that this distinction is primarily <u>logistical</u> and not technical. Field techniques can be applied at a fixed laboratory and complex analysis normally conducted in a fixed laboratory can be done in the field.

There are no differences in the quality principles that apply to analyses conducted at field or fixed laboratory locations. Examples of quality principles include, but are not limited to: QA, QC, procedure control, training, documentation, record keeping, corrective actions, self assessments and audits. There are differences in the procedures and practices used to follow the quality principles. The choice of appropriate quality procedures and practices is determined by the intended application of the resulting data and the methods selected to collect the data. *Hanford Analytical Services Quality Assurance Requirements Document* (HASQARD) differentiates between the intended data uses and establishes different quality requirements, procedures, and practices consistent with the analytical methods used to generate the data.

Most environmental activities at Hanford are directed towards regulatory application endpoints which include, but are not limited to: permit compliance, site or waste characterization, release of real property, site monitoring, site remediation, or site closure. There also are important information gathering efforts that support an environmental endpoint but they are not covered by regulation. This includes preliminary site surveys to support subsequent regulated characterization, release, or closure activities.   It also covers monitoring the progress of remediation activities by determining if the materials being remediated continue to exceed the agreed upon level for the "stop action" decision. It is unnecessary and costly to apply the quality requirements associated with data used to support regulatory endpoints to unregulated information gathering activities. Conversely, it is inefficient to apply the quality requirements related to information gathering activities to collect data that otherwise could be used to support an environmental endpoint. In many instances, data collected initially for the purpose of information can also be used to meet regulatory needs. Project planning activities must consider the most likely (highest valued) use of the resulting data when selecting between the less

demanding requirements associated with general information gathering and the more rigorous requirements associated with data used for regulatory purposes.

Full service laboratories are generally more able to provide an extensive range of quantitative analytical methods with their supporting equipment and infrastructure than are field laboratories. Traditionally, these laboratories are staffed and configured to support regulatory compliance activities. Field laboratories are generally equipped with limited analytical capabilities (methods), many of which are semi-quantitative. Field laboratories are also used as the base for *in situ analysis* by making use of portable equipment that can be brought directly to the sample or object being analyzed. Field analytical efforts at Hanford are generally directed to collection of preliminary site information or in support of remediation efforts seeking to determine if the site has reached agreed upon endpoints. As a general rule, fixed laboratory analyses are generally more sensitive than field analyses, in some cases take longer. Field analyses can provide quicker (shorter turn around time) and sometimes less costly analysis.

2.0   QUALITY ASSURANCE OBJECTIVES

QA objectives provide a set of recognized parameters to monitor and quantify performance of an analytical measurement. At Hanford, QA objectives are considered in the context of the overall objectives of the project that will use the data. The project and associated quality objectives are usually derived through the use of the data quality objective (DQO) process. The DQOs that result from the process are then translated into a sampling and analysis plan for the project.

This section provides a brief overview of DQOs and points out the role of the field analytical organization. For this document, the field analytical organization is any company or group whose primary services are radiochemical, organic, and/or inorganic analysis performed at the project site. Section 4.0 provides a more detailed discussion of the sampling and analytical planning activity that converts the DQOs into an implementation plan for the project. A discussion of the DQO process is found in Volume 1, Appendix A.

2.1   DATA QUALITY OBJECTIVES

The primary responsibility for identifying DQOs lies with the client or data user. The client or data user is also responsible for communicating this information to the organizations responsible for performing the work. The communication process should allow the organizations providing analysis in the field to understand and successfully meet client data quality requirements and comply with applicable regulations. Basic information about the nature of the sample(s) and the intended use of the data should be discussed and agreed upon by the field analytical organization and the client before sample collection. In the absence of a documented DQO process, as a minimum, the field analytical organization and the client shall agree upon the method performance criteria including precision, accuracy, and sensitivity for all analyses (e.g., instrument detectable limit [IDL], minimum detection level [MDL], minimum detectable concentration [MDC]). For field organizations performing *in situ analysis* (analysis of samples in place such as real-time air monitoring at set locations, radiological surveys of structures, equipment, or the environment), the client will identify critical locations for which analyses must be obtained.

The field analytical organization shall have a system to notify and explain to all staff performing work for the client any unique project requirements. Unique requirements are those that differ from the procedures described in this document and in the field analytical organization standard operating procedures.

The field analytical organization shall notify the client when situations that were not anticipated in the DQO documents and the sampling and analysis plans occur. These anomalies or nonconformances may result in plan changes by the project team. To the extent necessary, the field organization should make staff and information available to support the team to resolve the technical issues in a manner that meets the DQOs.

2.2   CLIENT DATA QUALITY REQUIREMENTS

DQOs are most often converted into a set of data quality requirements in the sampling and analysis plans (SAPs), quality assurance project plans (QAPjP), and other planning documents. There is usually more than one option about how the project may reach the desired certainty in project decisions. These are addressed in the last stage of the DQO process where an optimal SAP is selected by the project team. At this stage, the objectives are reduced to specific requirements including those for field analysis. The quality requirements shall be mutually understood and agreed upon by the field analytical organization and the client prior to the start of the field activity.

The client is responsible for ensuring access to the sample locations and that adequate sample material is available to meet their DQOs. This can be best achieved if the client includes appropriate *field analysis* professionals on the DQO team. If they are not included on the team, the analytical staff should review the DQOs, SAPs, or other planning documents as soon as possible to ensure that the field plans will meet the project objectives. The field analytical organization(s) providing *in situ analyses* and/or analyses in field laboratories is responsible for using proper *in situ* analytical procedures and protective sample handling protocols. The *field analysis* organization and the client share responsibility for selecting appropriate sample preparation and analytical techniques.

Five parameters are most often used to define project data quality requirements for the analytical portion of the plan. These are precision, accuracy, completeness, comparability, and representativeness. Of these, precision and accuracy are properties of individual data points that have a direct impact on data quality (see Volume 4, Section 6.0 for limitations associated with precision and accuracy). Comparability and completeness are properties of data sets that have an impact on data usability. Representativeness is a property of both data points and data sets. Individual samples or field measurements should be representative of the location where they were collected and data sets should be representative of the population being sampled. Taken together, these five parameters determine data quality and usability and should be considered when selecting a measurement technique.

The field organization has the responsibility to meet the precision, accuracy, and number of satisfactory data points (completeness) requirements which are based on the project error tolerances. This extends to both the sampling and the analytical effort. If the field analytical activity extends over time or requires that a number of different measurement techniques or instruments be used to measure the same property, the degree of comparability over time and/or method must also be established to ensure usability of the data set. The field organization is responsible to use proper sampling techniques (Volume 2, *Sampling Technical Requirements*) to collect unbiased samples from the target locations. The project team is responsible for the representativeness of the sample set which is determined by the unbiased nature of the sampling plan (location and number of samples).

The field organization should be informed by the project team if field analytical data (especially *in situ* measurements) may be used in supporting regulatory decision criteria. If so, the sampling and analysis plan must address the issue of comparability between the field methods and any methods that may be used to document, confirm, verify. or validate the final decision.

2.2.1   Precision

Precision represents a measure of the degree of reproducibility of measurements under prescribed similar conditions. Sample precision is calculated on the basis of duplicate analyses. Acceptance criteria shall be established for each analyte and each analyte method and shall be agreed upon by the field analytical organization and the client.

2.2.2   Accuracy

Accuracy represents the degree to which a measurement agrees with an accepted reference or true value. Sample accuracy is expressed as the percent recovery of an analyte in a reference material or a spiked sample. Acceptance criteria shall be established for each analyte and each analyte method and shall be agreed upon by the field analytical organization and the client.

2.2.3   Comparability

Comparability is the confidence with which one data set can be compared to another. When data are being collected over an extended period of time or using more than one instrument for measuring the same property of the target population, (i.e., identity of analytes, concentration of radionuclide), the comparability of the data depends on knowledge of the quantitative differences (in precision, accuracy, and sensitivity) that occur over time or from the use of different instruments. For each analyte, comparable precision and accuracy depend on the method and sample matrix. To be comparable, similar precision, accuracy, and sensitivity should be achieved on samples with similar matrices using similar methods. Factors such as analytical method detection limits or uncertainty, precision, accuracy, and matrix effects shall be considered when similar data are to be compared between multiple data collection activities. Furthermore, a statistically significant number of split samples or traceable standards should be used to establish comparability between different methods.

In many site characterization or site remediation situations, field methods are used as an indicator of the presence or absence (compared to a field decision level) of a radionuclide or other parameter of interest. In cases where the field measurement is used to estimate the actual concentration present in samples (based on more complex analysis) from a specific site or matrix, the two methods will be compared on a sufficient number of samples to develop a correlation curve. If a suitable correlation has been developed using an appropriate data set or from similar samples, the correlation need only be confirmed with a limited number of samples.

2.2.4 Completeness

Completeness is used to measure the amount of data produced by the sampling and analysis effort that meets the client's QC requirements (e.g., accuracy, precision, comparability). Care must be taken that the number of reliable field measurements meet or exceeds the number of measurements planned for in the DQO. This is necessary to determine if the DQOs were achieved in the sampling and analysis effort. If the level of completeness of the project data is lower than that needed to provide a statistically sufficient number of data points meeting the QC requirements, it may not be possible to determine if the DQOs were attained.

In some cases, data may not meet all the QC requirements but may still be used for qualitative information as an indicator of the presence or absence of a parameter. A clear definition of completeness allowed should be agreed upon by the field analytical organization and the client. Developing a requirement for critical samples that differ from other samples may also be useful.

2.2.5 Representativeness

Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, a parameter variation at a sampling point, a process condition, or an environmental condition. Representativeness of a population or an environmental condition depends heavily on sampling design and execution. Sampling design also covers the selection of site locations for *in situ analysis* conducted as part of field analytical activities.

The sample design is found in one or more of the following: the DQO documents, project SAP, Waste Analysis Plan, or other project planning document. Guidance in the implementation of the sampling plan is found in Volume 2.

Analytical data is not the sole indicator of representativeness of the sample or sample set. Proper subsampling techniques must be applied to samples collected in containers and later processed for analysis. If different phases or large variations in particle sizes are apparent in the sample, the analyst should consult with the planning documents or the client to determine if the variations were expected. The analyst should then determine if the sample handling instructions in the planning documents are adequate to preserve the representativeness of the analytical subsample. Any recommended changes or alterations to the plan should be discussed with technical personnel empowered to make decisions for the project. All changes to the plan must be documented in the report of analysis. *In situ analysis* does not introduce non-representativeness into the sample data set due to subsampling since the material being analyzed is not removed from the field.

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3.0   FIELD ANALYTICAL PLANNING AND DESIGN

This section discusses the use of semi-quantitative and quantitative analytical strategies as part of the design and planning of sampling and analytical activities. It also describes the six principle applications of analytical services in Hanford projects. The use of the graded QA/QC approach is recommended to match the analytical QA/QC requirements to the project need for semi-quantitative or quantitative data.

3.1   ANALYTICAL OBJECTIVES OF FIELD ACTIVITIES

Most field analytical activities fall into one of two categories:

1.   Semi-Quantitative Analysis—The detection and measurement of target analytes within a restricted or limited range of concentration or activity with a known degree of precision, accuracy, and/or sensitivity. Calibration addresses only the concentration (activity) range of interest (or a single point). QC is usually limited to confirming the calibration for the period in which analyses are performed.

2.   Quantitative Analysis— The detection and measurement of target analyte concentration or activity with known precision, accuracy, and sensitivity and a high degree of confidence. Calibration covers the full range of possible analyte concentrations (activities) in the samples of interest. QC procedures are sufficient to establish the analyte concentration (activity) in the sample matrix with the precision and accuracy (bias) necessary to meet the project requirements for overall confidence in the reported values.

A semi-quantitative or quantitative analytical strategy is selected during the planning phase to provide data sufficient to meet the project objectives. The project-specific DQO or other appropriate planning process establishes the objectives. The choice of which strategy is employed frequently depends on intended application of the data, which is related to the project life cycle (discussed in the next section).

The project planning team and the analytical organization shall agree upon the analytical methods, calibration, and QC procedures to meet the quality requirements for the analyses. Calibration and QC procedures should be selected using the graded approach to establish the technical defensibility and reliability (precision, accuracy, confidence) of the data while at the same time conserving time and dollar resources. Both types of analysis may be performed in either field locations (*in situ analysis* and *field analysis*) or in fixed laboratories providing all the requirements pertaining to the analysis are met.

3.2 EXAMPLES OF ANALYTICAL AND QUALITY CONTROL DESIGNS

Examples of the application of the two categories of analysis and the attendant calibration and QC are described below. These examples are for the purpose of illustration only. Actual applications must be based on project-specific requirements.

- Semi-Quantitative Analysis:

    — Site scoping using hand-held scanning devices to determine the absence of radionuclides below a pre-determined level. Calibration must establish that the measurement system is capable of providing a positive reading at the pre-determined level. Continuing calibration QC should be used at the beginning and end of each measurement day.

    — Waste characterization using lead test strips to determine the absence of lead in drums from a repository known to contain an unknown number of drums from a lead fabrication operation. A one-time demonstration that each batch of test strips gives a positive response to samples containing lead from a reference matrix similar to the one(s) resulting from the fabrication operation should be adequate to calibrate the procedure. A laboratory control sample (LCS) should be used once per day to demonstrate that the strips are functioning properly.

    — Site characterization using a hand-held hydrocarbon vapor monitor to locate soil areas containing spilled gasoline for more detailed analysis. A daily demonstration that the measurement system provides a positive response to a low-level reference vapor is sufficient calibration. This assumes that the low-level reference is below the level of concern for gasoline components in the vapor phase in soils. An LCS at a level greater than the calibration level and less than the level of concern should be run at the beginning and end of each measurement day. If the measurement system is unstable (hand-held, battery-operated optical devices frequently are), the LCS should be repeated on a batch-size basis to ensure satisfactory operation over long time periods (hours).

    — Remediation support activities using a portable x-ray fluorescence unit to establish that loads of contaminated soil exceed the cleanup standard for the area for inorganic constituents. The x-ray device should be calibrated at a level such that the number of false negative results meets the stakeholders' expectations. This should be determined once using a site-specific matrix and establishing the analytical variance of the technique at the acceptable clean up concentration and suitable statistical analysis. A daily QC sample (continuing calibration verification) should be used at the beginning and end of the analytical day.

— Verification of site status using a hand‑held radiological counting device to establish that no residual radiation above an accepted health‑based risk level remains on site. Calibration must establish that the measurement system is capable of providing a positive reading at the health‑based risk level. A one‑time comparison of the hand‑held readings with a reliable quantitative technique should be conducted to demonstrate the correlation of the hand‑held measurements with a recognized quantitative measurement system. In effect, this establishes a technique usually associated with semi‑quantitative analysis is reliable (in this application) for quantitative data. Continuing calibration QC should be used at the beginning and end of each measurement day.

— Waste characterization using polychlorinated biphenyl (PCB) analyses that apply immunoassay techniques to determine whether PCBs in oil are above or below 50 ppm. A one‑time demonstration that each reagent batch gives a positive response to samples containing PCBs above 50 ppm and below 50 ppm. Analysis of an LCS should be used once per day to demonstrate that the analysis is functioning properly.

— Characterization of headspace in a drum to monitor for flammable gases. The detector will see methane, hydrogen, ammonia, and other flammable gases. The detector is calibrated one time with several different gases at a level that exceeds 25% of the lower flammability limit (LFL) and a level that is below the LFL. A daily LCS check is performed to demonstrate that the concentrations above the LFL are detected.

• Quantitative Analysis:

— Site characterization for asbestos using polarized‑light microscopy to establish the level of all types of asbestos present in building debris. A certified analyst should demonstrate the ability to analyze certified reference materials on a daily basis. The type and frequency of QC samples must meet the regulatory requirements for the matrix.

— Waste characterization using EPA Method 1311 to determine if site waste materials are hazardous because of inorganic, volatile organic contaminants, or semi‑volatile organic contaminants. There are no calibration requirements for EPA Method 1311. Method‑specific QC requirements are as follows: one method blank per 20 uses of an extraction device (may be used as the method blank for a batch of client samples); one matrix spike added to the leachate prior to preservation (may replace the matrix spike required in the analytical method); for metals, the method of standard additions shall be used if the matrix spike recovery is less than 50% or if the measured concentration is $\pm$ 20% of the regulatory level. In addition, the calibration and QA/QC requirements of the various analytical methods must be met for the analysis of the waste and/or the waste leachates except as noted above.

— Verification of site status with respect to PCBs contamination using EPA Method 8082 to confirm that all areas of the site contain less than 2 ppm of PCB at the 95% confidence level. A single-point calibration may be used and it should be determined using a site-specific reference material, if possible. A sufficient number of replicates of the calibration standard should be analyzed to establish the analytical variance near the decision level. This is then used to determine the number of samples and the estimate of the mean PCB concentration (at the upper 95% confidence level) that demonstrates the release criteria of less than 2 ppm was achieved. The method-specific QC requirements and acceptance criteria are applied to all analysis.

— Characterization of the water from drain lines in a building planned for decommissioning and destruction for treatment. The characterization includes full gas chromatograph (GC)/mass spectrometer (MS) analysis of the volatiles and semi-volatiles, and 22 metals. The metals data by inductively coupled plasma (ICP) is used to assess hazardous levels of metals and whether precipitation of oxides will cause treatment problems. Normal QC is required per the SW 846 analytical method that includes multi-point initial calibration, daily calibration verification, method blanks, LCSs, and other method-specific QC such as surrogates for organics and interference checks for metals.

— Quantitative analysis of concrete cores are shaved in ¼-inch increments and analyzed for Cs^{137}, Sr^{90}, and $Pu^{239/240}$ to assess the isotopic distribution for use in risk modeling of concrete to remain buried below 15 ft. The quantitative analysis will be used to correlate to the survey data from the same core to develop a correlation with the Cs^{137}, gamma isotope of the portable detector. The portable device will be used for all subsequent measurements.

— Analysis of lead in paint is performed to determine whether building debris should be disposed as hazardous or non-hazardous waste. The same sampling and analysis could be used to assess whether an old building requires a fixative be applied to the walls before office workers use the building. The paint is shaved from the walls and analyzed by either ICP or atomic absorption to assess the lead content by SW-846 methods (EPA 1997) with the standard QC specified in the method.

More information about method-specific criteria for semi-quantitative and quantitative analysis is provided in Section 6.0, "Calibration" and Section 7.0, "Quality Control."

3.3   INFORMATION FROM THE PLANNING AND DESIGN ACTIVITY

The following sample/location-specific criteria shall be provided or agreed upon (as appropriate) and documented before the field analytical organization begins work:

- Applicable regulatory requirements such as sample location, chain-of-custody (if applicable), holding times, and QA/QC specifications.

- Site characteristics, process knowledge, sample source, and sample properties known to the client that could impact the worker safety.

- Handling of samples in the field, during transport, and in the analytical process.

- Estimated number and matrix of samples.

- Sample handling requirements relative to a specific sample or matrix.

- Analyte lists and procedures for sample analysis.

- Calibration range criteria.

- QC samples frequency, type, and acceptance criteria if different from those established in the approved analytical procedures.

- Correlation methods (if applicable).

- Expected dates of access to the field site, availability of special equipment or staff necessary to support the *field analysis* operation.

- Format, content of sample analysis reports, and format of electronic data deliverables (if requested).

- Turnaround time beginning from date of access to the field site for *in situ analysis* or sample receipt at the *field analysis* facility and ending with delivery of the report of results to the designated project person.

- Name, address, telephone number of client, and field analytical organization contacts responsible for the project, and information to establish electronic data transfer (e.g., type of software, file format).

- Methods for reporting, resolving, and documenting anomalies and nonconformances from expected site conditions and access to planned sample locations.

The following information shall be provided for samples that will be collected by individuals other than those performing the analysis for samples being analyzed at field laboratories: expected dates of sample receipt, sample preservation, delivery methods, storage and container types and volumes, holding times by method, and sample disposition requirements.

3.4   GENERAL OBJECTIVES OF FIELD PROJECTS

Experience at Hanford has shown that almost all environmental analytical activities can be described in terms of six different overall project objectives. These objectives are associated with the various phases of most field projects from initial site investigation through characterization and remediation to site closure and final verification of closure conditions. They are individual parts of the project life cycle and can be described as:

- site scoping,
- site characterization,
- waste characterization,
- remedial action support,
- site closure or final status surveys (FSS), and
- verification of status.

These phases in the life cycle of Hanford projects are generally consistent with the phases used in U.S. Department of Energy (DOE), Washington State Department of Ecology, and U.S. Environmental Protection Agency (EPA) programs that claim jurisdiction over most Hanford cleanup activities. The analytical work called for in each of the phases can be physical testing, radiological, organic, or inorganic analysis or any combination of the four. The analytical requirements are specified in the DQOs, SAPs, QAPjPs and other planning documents generated by the project team.

Each phase in the life cycle can be the subject of a dedicated project or part of a complex project activity covering more than one phase of the life cycle. Not all project sites go through all phases of field activity. The historical site assessment (HSA) that is conducted on most Hanford projects frequently provides sufficient information to meet the data requirements of many of the phases.

These various project data needs are the driving force behind the use of semi-quantitative or quantitative analytical strategies that result in a graded approach to QA and QC at Hanford. The use of the DQO process in which DOE, site contractors, and regulators agree, in advance, on the data needs ensures that only data to support project decisions are collected. The stakeholders also establish the quality requirements in the DQO process, which allow them to achieve their stated level of confidence in the final decision.

In general, each of these phases expands on the data collected during the previous phase (e.g., the characterization analyses are planned with information collected during the scoping phase or the HSA) up through the final status survey and project closeout. This is important to keep in mind

when preparing SAPs. Cost and time efficiencies may be realized for the project if the most demanding intended use for a data set is considered early in the project life cycle.

The purpose of the FSS or other project closeout analysis is to demonstrate that the release criterion and clean up criteria or cleanup criteria established by the regulatory agencies for radiochemical, organic, and inorganic constituents have not been exceeded. The responsible regulatory agency(ies) or their contractors usually carry out the final verification of site closeout. Therefore, it is important to know how the verification work will be conducted so that the closeout results (obtained by DOE or its contractors) are technically compatible with the verification results.

The following sections briefly describe the project objectives most common to the various phases of the project life cycle. As a matter of convention in these sections the term "site" is used to represent the land, building(s), or equipment that is being investigated.

3.4.1   Site Scoping

If the information collected during the HSA indicate a site is contaminated, a scoping survey/assessment may be performed. Scoping activities provide site-specific information based on limited measurements. Semi-quantitative measurements are frequently used in this application.

The primary objectives of a scoping activity are to:

- perform a preliminary hazard assessment to determine potential contaminants of concern (COCs),

- support division of the property into contaminated or uncontaminated (to a predetermined level) areas,

- provide input to the site characterization design (if necessary).

Site scoping activities are normally conducted after the HSA is completed and consist of judgmental measurements based on the HSA data. If the results of the HSA indicate that a property is uncontaminated at a predetermined level and no contamination above that level is found, the property may be classified as clean (to a "not to exceed level" for the constituents of concern). No further site characterization activity is needed and site closure measurements can be performed at the appropriate time to release the property for its intended use. If the site scoping activity locates contamination, the property may be considered as contaminated (above a predetermined level). A site characterization is typically performed next to confirm the location and level of contamination and collect data for selection of an appropriate remediation strategy. Site scoping data may provide input to the design of the site characterization phase.

Sufficient information should be collected to identify situations that may require immediate attention due to radiological or chemical contamination.

3.4.2   Site Characterization

Characterization may or may not be preceded by a scoping activity. If the historical or scoping data indicate a potential for chemical or radiation levels exceeding guidelines and are not sufficient to permit the preparation of a remediation or closure plan, characterization analysis is indicated. Characterization is based on the HSA and scoping results. This type of analysis provides a detailed environmental characterization of the land, building(s), and equipment. Quantitative analytical strategies are used more frequently than semi-quantitative ones during this phase.

The primary objectives of a characterization activity are to:

- determine the nature and extent of the contamination,

- collect data to support evaluation of remedial alternatives and technologies,

- evaluate whether the survey plan can be optimized for use in the FSS,

- support Remedial Investigation/Feasibility Study requirements (CERCLA sites only) or Facility Investigation/Corrective Measures Study requirements (*Resource Conservation and Recovery Act of 1976* [RCRA] sites only),

- provide input to the site closeout or FSS design, and

- as low as reasonably achievable (ALARA)[1] assessments.

Characterization analysis is the most comprehensive of all the data collection activities and usually generates the most data. This includes preparing a statistically sound sampling plan, making systematic, as well as judgmental measurements, and analysis of different media (e.g., surface soils, interior and exterior surfaces of buildings). The decision as to which media to examine is a site-specific decision addressed through the DQO process.

3.4.3   Waste Characterization

All hazardous Hanford wastes including radiologically contaminated material, organic and/or inorganic hazardous material, and mixed waste material must be treated or disposed of at

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[1]Describes the approach to radiation protection used by DOE to control or manage exposures to, and releases of, radioactive material. Its objective is to attain dose levels as far below the applicable limits of DOE Order 5400.5 (DOE 1993) as practical considering technical, economic, safety, and social factors.

permitted facilities. Waste characterization normally is performed to determine the level of potentially hazardous constituents in materials destined for treatment or disposal.

The waste characterization activity uses historical data, process information, and/or direct sampling and analysis of the waste material to determine if the waste is hazardous. Semi-quantitative data is collected most frequently when there is historical data and/or process information that define the waste with a high degree of confidence. The new data only needs to confirm the waste characteristic. Quantitative data is usually collected when the waste originates from an unknown source or uncharacterized location or for the verification of the waste characterization process.

If the waste is hazardous, it is transferred to the treatment, storage, and disposal (TSD) facility permitted to handle the material. The facility's waste acceptance criteria established in its operating permit determine if a particular waste can be shipped to the facility. While the primary objective of waste characterization is to determine if the material is hazardous, it may be prudent to use analytical techniques and QC requirements that meet the extended project needs to manifest the waste for shipment and meet the TSD facility's waste characterization criteria if the waste is expected to be hazardous.

3.4.4   Remedial Action Support

Remedial action support is normally analyses that are performed while land, building, and equipment decontamination is being conducted. The purpose is to provide an indication that the chemical and radioactive contaminants are actually being removed, to monitor the progress of the decontamination, and to verify that personnel are being adequately protected. Semi-quantitative analytical approaches are frequently suitable for this application.

Remedial action support is conducted to:

- support decontamination activities on real or non-real property (equipment),

- determine when a property or a section of property is ready for closeout analysis or the FSS, and

- provide updated estimates of property-specific parameters used for planning the site closeout analysis or FSS.

The determination that a piece of property is ready for a closeout analysis or FSS following remediation is an important step in the process. In addition, remedial activities result in changes to the distribution of contamination within the site. For most sites, the property-specific parameters used during closeout or FSS planning (e.g., variability in the chemical or radionuclide concentration, probability of small areas of elevated concentration or activity) will need to be re-established following remediation. Obtaining updated values for these critical parameters should

be considered when planning a remedial action analytical activity. Quantitative analysis may be the best choice for this special case.

3.4.5   Closeout Activities or Final Status Surveys

These activities are performed to provide sufficient data to demonstrate that the contamination has been removed (i.e., that the land, building, and equipment meets the criteria for release for appropriate future use or, where appropriate, designated restricted use) and that no unacceptable health risk exists. Closeout analytical plans and FSSs are detailed (i.e., use an existing grid or develop a new system, perform scanning, systematic soil sampling, and subsurface sampling) and essentially provide a new property characterization. However, the details should be commensurate with the need. The closeout activity demonstrates compliance with regulations. This type of analysis is the focus of the many Hanford environmental activities. The demonstration of regulatory compliance often requires the use of quantitative methods.

The primary objectives of the closeout activity are to:

*   select/verify site grouping,

*   demonstrate that the potential dose or risk from residual chemical contamination is below the release criterion for each site, and

*   demonstrate that the potential dose or risk from small areas of elevated activity or chemical contamination is below the release criterion for each property unit.

The closeout analysis provides data to demonstrate that all radiological and chemical parameters satisfy the established guideline values and conditions. Although the closeout phase is discussed as if it were an activity performed at a single stage of the property investigation process, this does not have to be the case. Data from other analysis conducted during the investigation and remediation process such as scoping, characterization, and remedial action support activities can provide valuable information for planning a closeout activity provided they meet project data requirements.

Professional judgment and biased sampling are important for locating contamination and characterizing the extent of contamination at a property. However, the closeout activity uses a more systematic approach to sampling. Systematic sampling is based on approaches that endeavor to achieve the representatives in sampling consistent with the application of statistical tests.

3.4.6   Verification Analysis

If the data suggest that the potential for contamination is low, or if the land, building, and equipment has been decontaminated and is ready for release, verification analysis is indicated.

Verification analysis confirms that all historical, characterization, remediation, and post-remediation data is adequate to demonstrate that the property meets applicable radioactive and chemical contamination release criteria. DOE typically recommends that this work be done by an organization that is independent of the contractor conducting the remediation to validate the accuracy and completeness of the field measurements and attest to the credibility of the cleanup and certification operations.

The regulatory agency responsible for the property often confirms whether the property is acceptable for release. This confirmation may be accomplished by the agency or an impartial party. Although some actual measurements may be performed, much of the work required for confirmation and verification involves evaluation and review of documentation and data from contractor activities.

The evaluation may include split-sample analyses by the regulatory agency's laboratory or site visits to observe survey and other analytical measurement procedures. Therefore, accounting for confirmation and verification activities during the planning stages is important to each type of analysis. In some cases, post-remedial sampling and analysis may be performed by an impartial third party. The review of results should include verifying that the DQOs are met, reviewing the analytical data used to demonstrate compliance, and verifying that the statistical test results support the decision to release the property. Confirmation and verification are generally ongoing processes throughout the project.

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4.0   SYSTEMS QUALITY ASSURANCE

A number of systems must be in place and operational when conducting *in situ* measurements and/or *field analysis*. These systems must function properly to produce and document the level of quality needed in the final product. These systems are the Administrative Systems, Software Systems, Technical Systems, and Physical Facility Systems.

4.1   ADMINISTRATIVE SYSTEMS

Volume 1 provides all requirements for the administrative systems that support field analytical activities

4.1.1   Documented Quality System

The field analytical team shall establish and maintain a quality system appropriate to the type, range and volume of sampling, calibration and/or testing activities it undertakes. The elements of this system shall be documented in a QA plan. The quality documentation shall be available for use by the field sampling and/or analytical personnel. The field analytical group shall define and document its policies and objectives for, and its commitment to, achieving quality in field analytical services. Management shall ensure that these policies and objectives are documented, communicated to and understood, and implemented by all field personnel concerned. The following topics shall be detailed in the documented quality system.

- Procedures for measuring data precision, accuracy, representativeness, comparability, and completeness. These details may vary on a project-specific basis but must be derived from project planning documents provided by the client. The procedures selected must be appropriate for proposed data use.

- Field operations including, but not limited to, sampling, special processes, purchased items and services, equipment maintenance, instrument calibration and use, field analyses, waste verification, geophysical surveys, and other site-characterization activities.

- Methods to be used in data reduction, validation, and reporting.

- Specific routine procedures used to assess data precision, accuracy, and completeness.

- Procedures for corrective action, nonconformance control, identification and control of items, and QC reports to management.

- A training and qualifications program shall be in place to ensure that all field sampling/analytical personnel in all positions have been trained and qualified in their specific area of work and also in the QA procedures associated with their work. The

program should be designed to ensure hat the training and qualification data are current and properly documented to ensure that suitable proficiency is achieved and maintained.

- Document control procedures to ensure that they are appropriate.

- Project interfaces and organizational structure.

4.2   SOFTWARE SYSTEMS

General requirements of Software Systems are discussed in Volume 1, Section 7.0. In addition, due to variances in using field portable equipment, the field analytical team shall ensure that data generated from instrumentation which uses a software system is backed up and/or downloaded on a regular basis. Any software configuration will require acceptance testing prior to use in the field.

4.3   TECHNICAL SYSTEMS

Technical systems assure that the techniques used are applicable and properly applied. These systems include sample exchanges, standards programs, control of standards and reagents, data reduction and reporting, data verification and validation, and technical audits/assessments. Procedures for documenting the above systems shall be established.

4.3.1   Field Assessments

During the actual performance of field activities, in-process self assessments or surveillances should be performed according to the requirements established in Volume 1, Section 10.2. At a minimum, one field assessment per year should be performed in each area of field activity. All assessments will ensure that the activities are being conducted according to approved procedures, and by qualified personnel. Results of assessments, including the corrective actions taken, should be reported to the project manager.

The audit/self assessment/surveillance of field analytical activities should evaluate, at a minimum, the following subjects:

- Equipment - Collection, measurement, and test equipment should meet the applicable standards (e.g. American Society for Testing and Materials [ASTM]) or have been evaluated as being acceptable to the procedures, requirements, and specifications.

- Verification of analysis activities that are the elements of the analysis program are in compliance with the applicable technical and quality standards, specifications and SAP requirements. The elements to be verified include, but are not limited to the following:

— Implementation of the QA Program.

— Qualification of personnel.

— Identification, control, and storage of samples, standards, project documents and generated data.

— Implementation of methods or procedures conforming to applicable specifications and SAP/WP requirements.

— Documentation and verification of conditions, observations and corrective actions taken.

- Completeness of records - examination of whether;

    — All measurements required by the QAPjP/SAP have been collected.

    — Complete records exist for each sample (or measurement) set and the associated QC samples.

    — The procedures specified in the QAPjP/SAP have been implemented and that changes have been noted according to the established procedures.

    — The results of the internal completeness check have been documented, and data affected by incomplete records have been identified.

    — Anomalous field data are identified and appropriately qualified.

- Evaluation of data with respect to control limits - Examination of corrective action reports to determine whether samples associated with out of control events are identified in a written report of the sampling activities.

- Review of holding time data - Examination of sample holding times in comparison to those required by the QAPjP/SAP. determine whether any samples associated with deviation from holding time requirements are identified in a written record of the activities.

- Implementation and effectiveness of corrective actions - Examination of corrective action reports to determine whether analysis activities associated with findings from previous audits/self assessments/surveillances of similar activities are identified in a written report. Determine if the facility response to previous findings was effective and properly implemented.

- Completeness and accuracy of reports to clients - The reports to clients will be examined to confirm that all data are accurately reported. This includes the reporting of QC data and its potential impact on data accessibility.

4.3.2   Field Documentation and Record Keeping

A records management system is required for both QA and investigation-specific records, which includes document control procedures, requirements for document review and records storage. Volume 1, Section 6.0, "Documents and Quality Records" describes the general requirements for this area.

For field analytical activities, records that provide direct evidence and support for necessary technical interpretations, judgments, and quality of work shall be specified, prepared, approved, and maintained. A procedure is required for field activity record keeping and document control. All data generated in the field associated with the end use of the data shall be maintained as quality records. Quality records include the following:

- Materials Certification: Documentation concerning the quality of material used on site shall be retained with the QA files and or project files. Examples of this type of documentation include:

    —   manufacturer and lot number for calibration standards and standard certificates of analysis,

    —   certificate of analyses for solvents used in decontamination (if applicable),

    —   certificates of cleaning or decontamination for commercially provided sample containers.

- Chain-of-custody.

- Field measurement data, calibrations, analytical results.

- Logbooks.

- Site-specific sampling plan, QAPjP, or work plan.

- Any sampling plan modifications or changes, field analytical procedural modifications, deviations or substitutions as well as the justification of the change and the precision and accuracy of the analytical method affected by the modification, deviation, or substitution.

All documentation must be completed in waterproof black ink. Corrections must be marked with a single line, dated, and initialed. Handwritten documents must be legible. All logbooks must be

permanently bound, individually identified with a control number, and water proof. The field logbooks are considered as the master record which documents the sampling event as well as for the operation and maintenance of all field analytical testing equipment

As a minimum, the following logbook entries will be made for each field analytical activity:

- names of personnel involved in the field activity;

- titles and responsibilities of personnel involved in the field activity (if applicable);

- signature of person making an entry (the printed name of the person is to occur at least once per logbook by their signature);

- type and purpose of field activity;

- site map, sketch, or other definitive site description;

- field observations;

- brief description of the job;

- sampling and/or field analytical methods used;

- instrument calibration information;

- Equipment identification numbers (if applicable);

- condition of equipment;

- field decontamination of equipment and personnel;

- documentation of equipment before arrival onsite;

- field problems, solutions, and corrective actions;

- attachments (e.g., photographs) with date of entry and initials of individual attaching, with a note identifying the attachment;

- audits or surveillances conducted during the sampling event;

- sample identification table with identification numbers, date, time, preservative, and analysis.

- field measurement data, types of method, types of QC samples used, and *in situ* data;

- field measurement location;

- lot numbers of the sample collection containers used;

- radiological screening information; and

- variances and field changes.

Field logbooks will be reviewed by team lead, person in charge or site appointed QA representative within a reasonable time frame to ensure that all applicable and necessary information is present to support the sampling and data quality.

4.4   PHYSICAL FACILITY SYSTEMS

4.4.1   Deployment

The physical layout of the site must allow deployment of a mobile laboratory, installation of monitoring equipment, or access by personnel to deploy hand-held monitoring equipment. The location and installation of monitoring equipment must ensure that the sampling component of the monitor is measuring a representative sample.

4.4.2   Power

Power is a major concern for *in situ* and or *field analysis*. Analytical equipment requires dependable power. Mobile laboratories require considerable power to operate analytical equipment. The preferred power source is line power. When line power is not available, generators shall be used. Power from generators requires conditioning before it is used to power analytical equipment. Running generator power through an inverter uninterruptible power supply is usually sufficient to produce clean line voltages and frequencies.

Power to data collection systems is also of concern. Signal level voltages are especially sensitive to fluctuations in supply voltages. Data quality can be severely compromised or rendered useless by random fluctuations in signal voltages. Field analytical equipment that is unprotected from the weather is subject to a wide variety of disturbances that can be disruptive to data collection and processing.

4.4.3   Weather Conditions

Weather conditions can peripherally affect *in situ* and *field analysis* measurements. Mobile field analytical laboratories are, in general, as resistant to weather fluctuations as a fixed laboratory. However, mobile laboratories need to be able to control their environment enough to minimize

the external effects that weather may cause. Protection of some sort is required for analytical equipment deployed outside. Generally monitors are housed in some type of enclosure that is reasonably weather proof.

Hand-held in situ, screening equipment is probably most affected by temperature fluctuations. Organic screening equipment will especially be affected. Changes in temperature will change the vapor pressure of the organic compounds, altering the amount of the compound the monitor will see. Care must be taken to calibrate the equipment under the same conditions that the samples will be analyzed under to minimize undesirable changes in calibrations of inorganic and organic compounds.

4.4.4 Facility Design and Maintenance

Proper facility design and maintenance can help alleviate problems associated with data generation. The following issues, at a minimum, should be addressed:

- Ventilation, with air exchange rates and pressure differential between work area, suitable working environment (e.g., lighting, temperature control), stable power sources and radio frequency shielding.

- Adequate space for field analytical activities so they do not adversely affect analyses. This includes solvent, standards, reagent, and waste storages, in addition to other work areas.

- Specialized equipment, such as an acid hood or glovebox, where required.

- Water purification.

- Preventive maintenance schedules for equipment.

- Proper maintenance to prevent contaminating vacuum systems.

- Storage of gases.

4.4.5 Field Analytical Laboratory Safety

Each mobile/field analytical laboratory shall maintain their operations in a safe manner. When *in situ analyses* are performed, portable eye-wash stations and appropriate spill kits shall be near the point of analyses. Entry into the mobile laboratories shall be limited to approved personnel. Whenever a mobile laboratory is performing analysis on radiologically contaminated samples, appropriate radiological working procedures shall be in place, as well as personnel monitoring, and in some cases, air monitoring. Entrances to the mobile facility shall have the appropriate signs on the entry door for personnel protective clothing and radiological conditions/controls.

4.4.6   Waste Management

Waste produced from *field analysis* can include sample media, calibration standards, syringes, empty sample containers, waste glassware, paper towels, gloves, and other expendable items. consideration should be given to reagents used to clean syringes or glassware, because used methanol, for example, is a listed hazardous waste (F003).

The handling and disposal of radioactive and hazardous waste generated by field analytical methods should comply with all federal, state, and local requirements. Procedures should incorporate controls that minimize the generation of waste and maximizes the concentration, recovery, and recycling of waste products to the extent economically practical. Site-specific requirements and responsibilities for waste handling and disposal of field analytical waste should be specified in the SAP or other project documentation.

Onsite *field analysis* has the benefit of generating a minimum volume of waste and often times should allow for the unaltered or unused sample to be returned to the sample point immediately following the *field analysis*. This practice should be used whenever possible for waste minimization.

Prior to selecting a field analytical method, an evaluation should be made to determine the characteristics and volume of any waste that may be generated as a result of the analysis. The evaluation should include a preliminary determination of whether the analysis will cause the generation of any hazardous, radioactive, and/or mixed waste. Additional consideration of potential listed waste is required, if applicable. Field screening methods should be selected in consideration of those that do not create hazardous waste.

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5.0   METHOD REQUIREMENTS

Field analytical services organizations must observe the requirements placed on procedures
covering analytical methods that are given in Volume 1, Section 4.0, "Procedures."

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6.0   CALIBRATION

In general, the performance of measurement systems is controlled through calibration and monitored by continuing verification of calibration. This section describes calibration activities associated with calibration requirements for field measurement systems and specifications for standards that are used for calibration.

Chemical measurements are made using systems that include sample handling and measurement processes. All aspects of the measurement process shall be calibrated. Ancillary data on such matters as temperature, pressure, humidity, particle size, volumetric capacity, mass, and flow rate may be needed as well, requiring accurately calibrated instrumentation for their measurement. Accordingly, any of the instruments, standards, and methods used for these purposes shall be calibrated to ensure that their accuracy is within acceptable limits.

The procedures used to calibrate instruments for radionuclide, inorganic, and organic analyses are based on the operating characteristics of the instruments used in the measurement process. The frequency of calibration is directly related to the stability of the instruments, especially the detector systems employed. Radiochemical detectors are extremely stable over time and may only require calibration annually or semi-annually. Some types of detectors used for organic analysis are much less stable and require calibration with each use. Volume 4, Section 4.0 discusses the calibration of instruments for the analysis of each type of chemical constituent (radionuclide, organic, and inorganic) individually. The various types of instrumentation used to analyze individual constituents within a type are also discussed.

This section discusses the calibration requirements for instruments commonly used in field applications at the Hanford Site. Most of the analytical techniques presented here are used for semi-quantitative analysis called out in project DQOs. This category of analysis is used in all six of the field applications discussed in Section 3.1 of this volume. The calibration and continuing calibration verification requirements are sufficient to meet the normal data quality requirements set out in the project planning documents such as DQOs. It is costly and ineffective to increase the frequency beyond that listed in the following sections. Conversely, less frequent calibration (and verification) may render the resulting data unsuitable for its intended use. Calibration requirements for quantitative analysis are found in Volume 4, Section 4.0.

Any changes to the calibration procedures or frequency that are required to meet special project DQOs shall be documented and communicated to the field measurement organization by the project manager before the start of any field measurement activity as required in Section 1.0.

6.1   CALIBRATION OF FIELD ANALYTICAL
MEASUREMENT SYSTEMS

The calibration process correlates instrument response to a reference concentration or characteristic. Calibration procedures shall be established by the field analytical organization

and shall consider the manufacturer's recommendations and the requirements specified in Volume 4, Section 4.0.

The initial calibration verification checks the accuracy of the calibration and the standards used for that purpose. A level of independence shall exist between the materials used for calibration and for initial calibration verification when such materials are available. When an independent source is not available, the field analytical organization should attempt to purchase an alternate lot of the same material.

The continuing calibration verification confirms that the original calibration is within acceptance criteria over time. This standard may be from the same source as that used for either calibration or initial calibration verification.

The requirements of calibration, frequency, acceptance criteria, and corrective actions for field analytical measurement systems are presented in Volume 4, Section 4.0, Tables 4-1 through 4-9. The field analytical group is required to take corrective action when measurement systems fail calibration QC criteria as demonstrated by the procedures discussed in Volume 4, Section 6.0,. As a general requirement, all analyses that were conducted using an instrument that fails the continuing calibration acceptance criteria shall be re-analyzed once the unit is restored to calibration. When recalibration is required at an unusual and/or increasing frequency, it may indicate a chronic problem with the measurement system and a more thorough corrective action investigation should be undertaken, see Volume 1, Section 5.0 for corrective actions.

6.2   CALIBRATION REQUIREMENTS FOR SEMI-QUANTITATIVE ANALYSIS

Semi-quantitative analysis requires that the instruments being used be initially calibrated according to the requirements presented in Volume 4 , Section 4.0. The calibration is usually performed over a very limited range of target analyte concentrations or activities or at a single level. The calibration must be confirmed by the use of an appropriate CCV standard (same level or activity as the initial calibration standard) at the beginning of each day when the instrument is used and as necessary during or at the end of the day.

Analytical instruments normally used for quantitative analysis can also be used for semi-quantitative analysis. The primary difference in the two is that in the quantitative application, the instrument is calibrated initially using a full range of calibration standards, a mid-range continuing calibration verification standard, more rigorous calibration acceptance criteria, and a higher frequency of CCV. Any instrument that is initially calibrated for quantitative analysis may be used for semi-quantitative analysis if the mid-range calibration standard is replaced by the single point CCV which meets the project-specific DQOs. The frequency of the CCV can also be replaced by that required for semi-quantitative determinations.

6.3   CALIBRATION REQUIREMENTS FOR QUANTITATIVE ANALYSIS

The requirements for calibrating measurement systems for quantitative analysis are found in Volume 4, Section 4.0.

6.4   CALIBRATION RECORDS

The field measurement organization shall keep a record of raw calibration data for all methods and instruments. Calibration records (initial calibration, initial calibration verification, and CCV) shall include the raw calibration data, associated reports, date of analysis, and analyst's name or initials, at a minimum. Calibration data shall be traceable to the standards used. All samples analyzed shall be traceable to the calibration under which the results were produced. Sample analysis can only proceed when measurement systems are accurately calibrated. All calibration records shall be maintained according to Volume 1, Section 6.0.

6.5   BALANCES, THERMOMETERS, AND PIPETTES

Calibration records of measurement devices such as analytical balances and thermometers for critical mass and temperature measurements shall be maintained. All analytical balances shall be calibrated annually, at a minimum, by an approved metrology organization. An approved metrology organization is one that has been evaluated and selected on the basis of specified criteria consistent with industry standards for the calibration of balances. These records shall contain the date of calibration, initials of the person performing the calibration, the identity of the device or serial number, and the date the calibration expires. This information shall be affixed on or near the balance. Acceptable balance calibration shall be verified and documented daily when in use. The accuracy of thermometers and thermocouples used for critical temperature measurements (e.g., refrigerator temperature for sample storage, total dissolved solids analysis) shall be verified annually by comparing readings of such devices with the readings of a National Institute of Standards and Technology traceable factory-certified thermometer. If radiological conditions limit this capability, then the thermometer should be checked at the ice point.

It is considered good practice that mechanical pipettes used for critical measurements be verified to ensure acceptable performance. Before use, single-delivery volume checks should be performed and documented.

6.6   GENERAL REQUIREMENTS FOR CALIBRATION STANDARDS

The following standard specifications shall be used unless otherwise specified in Section 4.4 of this volume.

Standards used for calibration of measurement systems shall be traceable to a nationally or internationally recognized standard agency source or measurement system if available. A program for verifying and documenting the accuracy and traceability of all working standards against appropriate primary grade standards or the highest quality standards available shall be routinely followed. Complete documentation of the standards shall become part of the permanent record of the project.

Standards used for calibration shall be accompanied by a certificate or record that includes the vendor, lot number, purity, date of preparation and/or expiration, and concentration or activity of the standard material. At a minimum, the following information shall be maintained about standard preparations and if possible placed on the label:

- name of preparer,
- date prepared,
- standard identification number,
- dilution performed,
- final concentration or activity,
- expiration date or shelf life (if applicable).

When recognized standard material is unavailable or its purchase is impractical, the field analytical organization should attempt to purchase standard material from a reliable source. The field analytical organization shall have procedures in place to determine the acceptability of such materials.

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7.0   QUALITY CONTROL

7.1   INTRODUCTION

The purpose of this section is to provide QC guidance and establish requirements for field
analytical methods to ensure that data meet the DOE objective for technically defensible data
that meet project needs. The material presented here focuses on semi-quantitative analytical
applications. Similar information is presented in Volume 4, Section 6.0 for quantitative analysis.

QC is defined as the overall system of activities that provides checks and balances to ensure the
quality of a product or service. QC data are generated during the conduct of analytical activities
and evaluated against client requirements to measure how well the data meets the client's needs.
If the quality requirements are not being met, corrective action must be taken to bring the
measurement process under control to meet the DQOs. The goal is to provide technically
defensible data of known quality that is adequate for the client's intended purpose as expressed
in the project DQO or other planning documents. QC data allows the field analytical group to
take necessary corrective actions during the analytical process. After the work is complete, QC
records demonstrate the degree of conformance against specific requirements.

QC samples are incorporated throughout the sample collection and analysis processes to provide
data for evaluating the effectiveness of analytical processes. QC samples permit assessment of
the quality of field-generated environmental data. The information gained from the QC
assessment can then be used, where necessary, to implement corrective actions during the
analytical process or to improve processes for future application.

The field analytical group responsible for *in situ* and *field analyses* must ensure that the QC
applied to a given scope of work is capable of meeting client objectives for selectivity sensitivity,
precision, and accuracy, or the field analytical group must negotiate alternative requirements.

7.2   OVERVIEW OF QUALITY CONTROL
PROCEDURES

7.2.1   Graded Approach to Quality Control

The HASQARD QA/QC program is based on a wide variety of QC samples. The results are
used to measure method performance, guide real-time corrective action, and document the
reliability of analytical data. These QC procedures apply to both quantitative and semi-
quantitative analysis. However, the requirement for specific QC procedures is guided by the
DQOs of the individual project. The only requirements that apply to all measurements is that the
measurements system be responsive to the target analyte in known manner (frequently referred
to as calibration) and that this response be confirmed at intervals during the analytical process
(continuing calibration verification). All other QC procedures evaluate specific aspects of
method performance and execution. These procedures are applied as often as needed to ensure

that variables known to effect method performance are in control during the analysis. These are the procedures that must be identified during the DQO or other type of planning process.

Method performance is usually expressed in terms of selectivity, precision, accuracy, and sensitivity.

The major variables affecting analytical performance and subject to QC are:

- type of measurement system (instruments) used,
- analytical method used,
- sample matrix,
- target analytes,
- time,
- differences between operators, and
- differences between laboratories.

The HASQARD QC requirements establish controls to ensure a documented degree of precision, accuracy, and reproducibility. Based upon the experimental design, QC samples allow the data user, working in cooperation with the analytical organization, to control and document analytical performance based on data needs, the characteristics of the site, and the analytical resources available.

If no quality requirements are established by the data user and transmitted to the field analytical organization in work authorizing documents, the field analytical organization shall perform the requirements as specified in the following sections.

7.2.2 Field Analytical Requirements

Tables 7-1, 7-2, and 7-3 establish which QC samples are required for semi-quantitative and/or quantitative analysis. The tables also present a brief definition and description of the purpose of each sample. The tables are organized according to instrument, method, and sample matrix QC. Those QC samples that are applicable to a specific class of analytes (radiochemical, organic, and inorganic) are also indicated.

For those QC procedures not required for semi-quantitative analysis, the potential value added is pointed out for project-specific applications where a higher level of confidence may be desired than is provided by the required QC. These samples are indicated as "Recommended" in the tables. Section 7.2.3 provides guidance where these samples may offer significant value-added to under specific field applications.

The instrument QC samples listed in Table 7-1 evaluate the performance of the instrument (measurement system) under ideal conditions and/or with ideal matrices and target analytes. The method QC samples given in Table 7-2 evaluate possible impacts from the method (e.g., sample preparation, reagents, temperature, and execution). The matrix QC samples presented in

Table 7-3 evaluate the impact of other constituents or the sample matrix in the field sample in the measurement of the target analyte.

As pointed out in the previous section, the proper experimental design will allow the analyst and data user to determine the level of performance of analysis across any of the variables listed in Section 7.2.1. The specific QA/QC criteria that are applied to any set of analyses shall be decided by the project team during the planning process and communicated to the laboratory in work-authorizing documents.

7.2.3 Guidance in the Use of Recommended and Optional Quality Control Standards in Semi-Quantitative Analysis

Tables 7-1, 7-2, and 7-3 list many of the QC procedures associated with instrument, method, and matrix QC respectively, as recommended or optional for semi-quantitative analysis. These are the QC procedures that should be considered by the data user and the laboratory for inclusion in analytical sequence when certain field conditions exist or when an additional level of QA/QC is considered important. This is the graded approach to QC.

Table 7-4 provides guidance on the potential value-added for the various QC samples in semi-quantitative applications where there is not a requirement for the procedure.

7.3 FREQUENCY OF QUALITY CONTROL SAMPLES ANALYSIS

This section provides guidelines on the application and use of QC samples in batches of samples. A batch is typically defined as 20 or fewer of the same or similar matrix. In some analytical applications, batch size may be increased. The choice of which of these QC samples to use is discussed in Section 7.2.

7.3.1 Blanks

It is recommended that one method blank, where applicable should be analyzed per batch of 20 or fewer samples. Equipment, trip, and field blanks shall be analyzed as required by project-specific QA plans.

Table 7-1. Instrument Quality Control Procedures.

QC Procedure	Definition	Purpose	Use (by Analytical Category) Semi-Quantitative	Quantitative
Instrument Blank	A solvent or reagent blank.	Establishes instrument baseline.	Recommended	Required
Initial Calibration	A series of standards containing target analytes at different concentrations.	Defines the retention times, linearity and dynamic range of the responses of compounds of interest for a specific instrument and detector.	Required	Required
Continuing Calibration Verification (organic, inorganic, radiochemical)	A calibration standard containing target analytes at an appropriate concentration introduced and re-analyzed during the course of sample analysis.	Verifies the instrument is still performing within the initial calibration.	Required	Required
Interference Check Standard (ICP, ICP/MS)	A standard with known concentrations of elements that interfere with the analysis of target analytes.	Accounts for the affects of matrix constituents on the results of the analysis of target analytes.	Required	Required
Performance Evaluation Mixture (pesticides/GC)	A standard containing eight specific pesticides compounds.	Establishes the extent of breakdown of Endrin and 4,4"-DDT in the gas chromatographic injection and column system.	Optional	Required
Resolution (organic, inorganic)	A standard containing the target analytes.	Optimizes measurement precision and accuracy.	Optional	Required – (method specific)
Tune (organic GC/MS)	Standard consisting of one organic compound.	Ensures correct instrument mass calibration, mass resolution, and mass transmission.	Required	Required
Background Analysis (radiochemical)	Data acquisition without an actual sample.	Establishes the variable background contributions to the instrument signal.	Required	Required
Energy Calibration Check (radiochemical)	A standard containing an isotope with known energy.	Verifies the energy range of an isotope.	Optional	Required

GC   =   gas chromatograph.
ICP =   inductively coupled plasma.
MS   =   mass spectrometer.
QC   =   quality control.

Table 7-2. Method Quality Control Procedures.

QC Measures	Definition	Method QC — Purpose	Use (by Analytical Category) Semi-Quantitative	Use (by Analytical Category) Quantitative
Laboratory Control Sample	A sample of known composition, prepared and analyzed like a field sample.	Determines the accuracy of the analytical process without interferences from the sample matrix.	Optional	Required
Method Blank/Preparation Blank	A blank free of analyte and containing all reagents, internal standards, and surrogates carried through entire analytical process.	Accounts for any interference caused by the method reagents.	Optional	Required
Internal Standards (ICP, ICP/MS, GC, GC/MS)	Compounds of the same chemical class added to the sample extract just before analysis.	Determines response factors of standards as a basis for quantitation of target analytes.	Optional	Required
Surrogates (organic)	Compounds similar to target analytes in chemical composition, extraction, and chromatography, but very unlikely to be present in the field sample; carried through the analytical process.	Estimates overall recovery of the analytes of interest in the sample preparation and analysis process.	Optional	Required
Duplicate	The second preparation and/or analysis of a field sample.	Determines the precision of the analytical process for a particular matrix.	Recommended	Required
Retention Time Check	A standard containing the target analytes. The continuing calibration verification sample can be used for this purpose.	Confirms retention time of target analytes.	Optional	Required

GC   =   gas chromatography.
ICP =   inductively coupled plasma.
MS   =   mass spectrometry
QC   =   quality control.

Table 7-3. Sample Matrix Quality Control Procedures.

QC Measures	Definition	Purpose	Use (by Analytical Category) Semi-Quantitative	Use (by Analytical Category) Quantitative
		Sample Matrix QC		
Duplicate	The second preparation and analysis of a field sample.	Determines the precision of the analytical process for a particular matrix.	Recommended	Required
Matrix Spike (organic, inorganic)	A field sample spiked with the target analytes.	Assesses the effects of the matrix on recovery of target analytes.	Optional	Required
Matrix Spike Duplicate	A duplicate of Matrix Spike.	Determines the precision and accuracy of the method in the matrix.	Optional	Required
Post Digestion Spike (metals)	A field sample that has been digested and spiked with the target analyte compounds of that specific analysis.	Determines if low-spike recovery from a matrix spike sample is caused by matrix interference.	Recommended	Required
Serial Dilution (ICP/metals)	A five-fold dilution of a field sample when analytes exceed the calibration range.	Determines if high readings are due to matrix effects or actual elevated levels.	Optional	Required
Tracers (radiochemistry)	An isotope, unlikely to be present in the field sample, added to the sample and carried through the entire analytical process.	Quantifies the level of correction needed in the determination of the isotopes of interest because of loss during preparation.	Optional	Required
Surrogates (organic)	Analytical compounds that are similar to analytes of interest in chemical composition, extraction, and chromatography, but very unlikely to be present in the field sample; carried through the entire analytical process.	Estimates the overall recovery of the analytes of interest in the sample preparation and analysis process.	Optional	Required

ICP = inductively coupled plasma.
QC = quality control.

Table 7-4. Potential Value-Added for Gas Chromatograph in Semi-Quantitative Analysis. (3 Pages)

QC Procedure	Purpose	Use in Semi-Quantitative Analysis	Potential Value-Added
Instrument QC			
Instrument Blank	Establishes instrument baseline.	Recommended	Some instruments have built in procedures for re-establishing the baseline automatically or manually between readings. This occurs normally during the analytical operation and should be addressed in the method SOP and the procedure should be used in all applications. For instruments that have detectors subject to drift such as photomultiplier tubes, it may be necessary to re-establish the baseline (and re-calibrate) on a regular basis to maintain QC and avoid re-analysis.
Performance Evaluation Mixture (pesticides/GC)	Establishes the extent of breakdown of Endrin and 4,4"-DDT in the gas chromatographic injection and column system.	Optional	If the sample matrix contains high levels of organic materials that are introduced into the GC injector region and on the column, a performance evaluation sample should be run after any calibration verification sample that fails to meet acceptance criteria. This will eliminate the use of a system with declining performance due to fouling leading to more stable calibration.
Resolution (organic, inorganic)	Optimizes measurement precision and accuracy.	Optional	Satisfactory resolution of the GC and GC/MS system for the target analytes should be confirmed for matrices containing high levels of co-eluting material (oils or interfering compounds) that may interfere with automated data collection/reduction. This will permit the implementation of advanced automated methods or manual methods for quantitation and improve analytical accuracy and/or prevent continuing calibration failures that are caused by the matrix.
Method QC			
Laboratory Control Sample	Determines the precision of the analytical process without interferences from the sample matrix.	Optional	For methods/samples requiring a preparation step, confirms the variation of between-sample results is real and not related to preparation/analyst performance. For methods/samples that are measured directly without preparation, the continuing calibration sample provides comparable information.
Method Blank/ Preparation Blank	Accounts for any interference caused by the method reagents.	Optional	Can prevent false positive conclusions. Confirms that positive responses are sample-related and not due to solvents, equipment, or reagents used in the analysis. Has increasing value as the level of concern (DQO) approaches the lower limit of method reliability. Also has value when the target analytes are found at the site/laboratory in areas not associated with the target sample (site or laboratory introduced contamination).
Internal Standards (ICP, ICP/MS, GC, GC/MS)	Determines response factors of standards as a basis for quantitation of target analytes.	Optional	Can reduce the occurrence of inaccurate data if the sample matrix contains co-eluting interferences that impact the correct response of the internal standards used in the quantitation of target analytes.

Table 7-4. Potential Value-Added for Gas Chromatograph in Semi-Quantitative Analysis. (3 Pages)

QC Procedure	Purpose	Use in Semi-Quantitative Analysis	Potential Value-Added
Surrogates (organic)	Estimates overall recovery of the analytes of interest in the sample preparation and analysis process.	Optional	For methods/samples requiring a preparation step, confirms the variation of between-sample results is real and not related to preparation/analyst performance. For methods/samples that are measured directly without preparation, the continuing calibration sample provides comparable information.
Duplicate	Determines the precision of the analytical process for a particular matrix.	Recommended	Confirms the assumptions made in the DQO about the precision of the analysis. Knowledge of method precision can be used to estimate overall confidence in decisions based on population characteristics (median value, average value, highest/lowest value, etc.) from a number of measurements. Can lead to reduction in the number of data points collected (better precision than estimated in the DQO process) or an increase to achieve level of confidence desired by project team (worse precision than estimated). This has considerable value-added if the semi-quantitative method is being used as a quantitative estimator (after appropriate method correlation). Should probably be made a QC requirement in this application
Retention Time Check	Confirms retention time of target analytes.	Optional	Has little added-value in samples free from interferences or instrument degradation. Similar data can be gained from each sample and the continuing calibration verification samples.
Sample Matrix QC			
Duplicate	Determines the precision of the analytical process for a particular matrix	Recommended	Confirms the assumptions made in the DQO about the precision of the analysis in sample matrix. Knowledge of method precision can be used to estimate overall confidence in decisions based on population characteristics (median value, average value, highest/lowest value, etc.) from a number of measurements. Can lead to reduction in the number of data points collected (better precision than estimated in the DQO process) or an increase to achieve level of confidence desired by project team (worse precision than estimated). This has considerable value-added if the semi-quantitative method is being used as a quantitative estimator (after appropriate method correlation). Should probably be made a QC requirement in this application.
Matrix Spike (organic, inorganic)	Assesses the effects of the matrix on recovery of target analytes.	Optional	Can detect the occurrence of unexpected matrix effects. Has the most value-added when the variation in the sample matrix is considerable over the range of samples comprising the analytical batch or analytical day. Can minimize the number of false positives/ negatives.
Matrix Spike Duplicate	Determines the precision of the method in the matrix.	Optional	See above. The matrix spike duplicate documents the precision of data across the range of matrices in the analytical batch or analytical day.

Table 7-4. Potential Value-Added for Gas Chromatograph in Semi-Quantitative Analysis. (3 Pages)

QC Procedure	Purpose	Use in Semi-Quantitative Analysis	Potential Value-Added
Post Digestion Spike (metals)	Determines if unacceptable spike recovery from a matrix spike sample is caused by matrix interference with sample preparation step.	Recommended	Can detect the occurrence of unexpected matrix effects on the sample digestion step in metals analysis. Has the most value-added when the variation in the sample matrix is considerable over the range of samples comprising the analytical batch or analytical day. Can minimize the number of false positives/negatives.
Serial Dilution (ICP/metals)	Determines if high readings are due to matrix effects or actual elevated levels.	Optional	Has limited value-added if data objective is pass/fail about a decision point. Will have more value-added if accurate estimate of high concentrations assists in future planning.
Tracers (radiochemistry)	Quantifies the level of correction needed in the determination of the isotopes of interest because of loss during preparation.	Optional	Failure to correct for tracer recovery can lead to false negative results. Has increasing value–added as the analyte concentration approaches the decision level. Has declining value as the simplicity and reliability of separation step increases.
Surrogates (organic)	Estimates the overall recovery of the analytes of interest in the sample preparation and analysis process.	Optional	Can minimize false negative responses in cases where significant losses occur during sample preparation. Has the most value in complex matrices that present procedural challenges to sample preparation.

DQO = data quality objective.
GC = gas chromatograph.
ICP = inductively coupled plasma.
MS = mass spectrometer.
QC = quality control.
P = Procedure.

7.3.2   Standards

After the stabilization and calibration of the analytical equipment in accordance with the analytical method, the calibration shall be verified with the analysis of an independent verification standard. A qualified person different than the analyst should prepare an acceptable independent standard, or it can be purchased as a certified pre-made standard from a vendor. Laboratory-prepared verification standards are preferably made with chemicals from a different source than the chemicals used in the calibration standards. If the independent verification fails to pass the method-specific acceptance criteria, the calibration sequence must be repeated.

A CCV is analyzed to show that an instrument or method is in control during the analysis. The CCV shall be performed at the beginning and end of the analysis for semi-quantitative analysis. The CCV can be one of the standards from the initial calibration curve. The CCV must meet the method-specific acceptance criteria or re-calibration must be performed. Any samples not bracketed by compliant verification standards shall be reanalyzed.

Performance check (i.e., bromofluorobenzene [BFB], performance evaluation mixture, difluorotriphenylphosphire [DFTP], energy calibration check) standards are analyzed in accordance with the instrument-specific criteria as stated in instrument-specific procedures or manufactures criteria. Performance check standards shall meet the QC specified in the instrument-specific procedure before analysis can begin or continue.

Internal standards and surrogates shall be added to all samples, standards, and blanks as appropriate.

7.3.3   Duplicate Analysis

A duplicate analysis of one sample per batch of 20 or fewer samples shall be performed. Depending on the analytical methods, this can be a sample duplicate or a matrix spike duplicate.

Laboratory duplicates are two aliquots of the same sample (intralaboratory splits) that are taken through the entire sample preparation and analytical process. Spike duplicates are two spiked aliquots of the same sample that are taken through the entire sample preparation and analytical process. Duplicates are used to assess the precision of the preparation and counting process in a client-specific matrix. Agreement between duplicates indicates the reproducibility (precision) of the combined preparation/separation, and measurement process.

Disagreement can occur due to analyte concentration differences within the sample matrix (non-homogeneity) that are not amenable to analyst control during the analytical process (e.g., isolated particles of analyte in a soil matrix which cannot be reliably sampled using standard laboratory subsampling techniques). Disagreement may arise if the method has poor applicability to the analyte/matrix system.

In cases where the sample is not expected to contain concentrations of target analytes sufficient to produce relatively small measurement errors, the use of a matrix spike and matrix spike duplicate should be considered.

Radiochemical duplicate relative percent difference criteria are set at 20%. This criteria shall only be applied to an analyte concentration or activity which has an uncertainty (1 sigma) less than or equal to 15%. When either the sample or duplicate uncertainty exceeds 15%, the data shall be evaluated based on statistical comparability. The laboratory can also perform an evaluation using the mean difference. The mean difference calculation takes the uncertainty of each individual measurement into account when comparing the two results. In those cases where the criteria above are not met, evaluation of the source of error and impact on client data requirements shall be performed. When client data requirements can not be met the client shall be notified; results shall either be accepted or new work scope (methodology) agreed upon.

7.3.4   Matrix Spike

In general, a matrix spike is a client sample that has been spiked with the analyte(s) of interest and processed in the same manner as the sample. The matrix spike is used to monitor method performance in a specific sample matrix. Matrix spike results are indicators of the effect the client sample matrix has on the accuracy of measurement of the target analytes. If required by the method, one spike sample per batch shall be analyzed.

In radiochemistry, the matrix spike represents the addition of a known quantity of the isotope of interest to an aliquot of sample. This spike may be added to a sample aliquot prior to any sample preparation (i.e., fusion, leaching). Alternatively, it may be elected prior to specific radiochemical manipulation (e.g., separation chemistry or evaporation onto a planchet).

The decision on when and how to spike a sample is based on the anticipated sample analyte activity/concentration or required dilution. Spiking a sample that already exhibits high activity or concentration is not justifiable, either because of standard material consumption or radiation dosimetry issues. Likewise spiking before a large dilution can waste expensive standard material. Therefore, spiking may be performed after preliminary sample preparation and dilution, but before any separation or digestion occurs. However, spike amounts should always provide results exceeding the decision or action limit.

Each field analytical organization shall evaluate matrix spike recovery information against client data quality requirements. The goal is to ensure that limitations on the data caused by the sample matrix and represented by matrix spike performance, are adequately portrayed and discussed in the report to the client. It should be noted that client radiochemical results would already be corrected for matrix and/or handling effects if a tracer or carrier is used in place of a matrix spike.

When the analyte concentration is unknown, spiking is typically performed at a level that is one of the following: (1) equivalent to the threshold established by the DQO process, (2) specified

by method, or (3) one to five times the minimum detectable activity (MDA)/MDC. Otherwise, (as general guidance) the spiking should be performed at a level equivalent to one to five times that of the sample. In those instances where the analyte concentration significantly exceeds the amount of spike added in the prepared samples, the data must be further evaluated to determine if measurement of the spiked sample is statistically significant. If not, a re-run of the process may be required depending on client needs. Matrix spike control is demonstrated when target analytes are within established control limits. Control limits are established by one of the following: (1) established by the DQO process for a particular project or program or (2) laboratory performance over time in samples of similar matrix and concentration levels.

A matrix spike shall be prepared with each batch of samples processed together and the results reported to the client along with the calculated recovery. No adjustment of the client sample results is made in the laboratory report.

7.3.5 Instrument-Specific Quality Control

Most of the analytical methods used in quantitative analyses have instrument-specific QC requirements. Table 7-1 includes more details. In all cases, the analyst should demonstrate that the method is in control prior to performing sample analysis. If these instrument-specific QC requirements are not met before or during analysis, the analysis shall be terminated and the source of the problem identified and corrected.

7.4 GENERAL FIELD ANALYTICAL QUALITY CONTROL

In this section, QC requirements for collection and analysis of samples using radiochemical, inorganic, and organic analytical procedures are described. The use of non-certified reagents and standards, materials, and equipment and methods of lesser quality can result in added interferences, lesser precision and accuracy. These conditions are likely to be encountered in field applications. Each field analytical group shall have a mechanism in place for demonstrating control over such sources. A list of sources is described in the following sections.

7.4.1 Distilled or Deionized Water

High-purity water is generally defined as water that has been distilled or deionized, or both, so that it will have conductivity less than 1.0 mho/cm (greater than 1.0 megaohm-cm resistivity). Each field analytical group shall ensure that water used for data collection activities is of sufficient quality for the operation performed. Water quality shall be regularly monitored through analysis of method blanks when method blanks are required.

7.4.2   Compressed Gases

Each field analytical group shall monitor the quality of gases used in the field instruments to ensure that they are adequate for the operation being performed. At a minimum, this shall consist of monitoring system performance (e.g., for contribution to background and/or blanks from impurities).

7.4.3   Reagents

Each field analytical group shall use reagents for data collection activities that are of sufficient quality for the operation performed. Reagent quality shall be regularly monitored via preparative and analytical QC performance.

7.4.4   Labware

Each field analytical group shall purchase and use labware of sufficient quality to meet client requirements. Labware selected shall be compatible with the testing performed.

7.4.5   Glassware Cleaning

Glassware cleaning shall be performed in a manner that minimizes sample contamination.

7.4.6   Good Housekeeping

Each field analytical group shall maintain their operations in a clean and organized manner to maximize available workspace and minimize environmental impacts on sample quality.

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8.0 DATA COLLECTION, REDUCTION, REVIEW, AND REPORTING

In general, data collection and reporting processes encompass proper sampling, accurate chain-of-custody, proper collection of raw data, accurate data reduction and calculation, and the precise transfer of results to a final form. After the data is collected, reduced, and reviewed, it is reported to the client in an easy useable form. A copy of the report to the client and all supporting analytical information used to generate the report are then assembled and archived as part of the permanent project record. Volume 4 details the requirements and provides guidance for the specific procedures as they are applied in the fixed laboratory application. This section in Volume 3 discusses how these processes are applied to *in situ* field analytical applications and *field analysis* in a field laboratory.

All of the records described in this section shall be maintained in a traceable manner as part of the permanent project file to safeguard the data and meet regulatory requirements as described in Volume 1, Section 6.0. This will permit the reconstruction of all relevant activities that produced the reported data.

8.1 DATA COLLECTION

Raw data are all parameters used to calculate a final reportable result. Raw data can be generated by manual and/or electronic means. Manual data generation records shall be collected and documented according to applicable procedures. *In situ analysis* makes frequent use of field logbooks and forms for real-time data collection. Some *in situ* field measurement instruments may electronically record field information that is later downloaded or otherwise transferred to more permanent forms of storage. *Field analysis* performed at an onsite laboratory frequently captures manually generated data in logbooks or as electronic records that are not interfaced to central data storage devices used in fixed laboratory applications. Procedures shall be in place that detail how these field records are captured and protected as part of the permanent project file.

Many instruments are interfaced with computers and/or integrators and are able to generate and/or reduce the raw data into reportable results. Procedures shall be in place to describe the use of automated instruments. The procedures will address the processing of raw data for reporting to the client and the use of instrument-generated reports that are transmitted to the client. To the extent practical, both forms of data, manually calculated and instrumentally generated, should be reported to the client in the same format with the same number of significant figures when the two sources of data are used to meet the same information requirement as specified in the DQOs, SAP, or other planning documents.

Entries into logbooks shall be made in a manner such that they can be easily read, understood, and reproducible with a standard photocopier. Indelible ink shall be used. Corrections shall be made by drawing a single line through the erroneous entry, and initialing and dating the correction.

Raw data output shall be retained as a part of the records (see Volume 1, Section 6.0). Information on the dates of *in situ analysis*, sample collection, sample preparation and analysis; sample identification numbers; analyst or instrument operator; type of analysis; and procedure number including the revision number, shall be traceable to the raw data output.

8.2   DATA REDUCTION

Data reduction is defined as those mathematical operations applied to the raw data to produce a final reportable result. Data resulting from analyzing samples shall be reduced according to applicable procedures. Data reduction includes activities that convert instrument and computer responses into reportable results. These activities may involve calculations, changes to the units or the data values, and statistical and mathematical analysis.

Computer programs or spreadsheets used for data reduction shall be verified before reporting data to ensure data calculation and manipulation programs perform properly. This applies to those programs that are embedded in instrument-specific software/hardware packages as well as those that operate on data entered into stand-alone data reduction routines created by third-party vendors or Hanford Site personnel.

The following practices shall be established in the laboratory's procedures or QA plan to ensure accuracy of data entry, proper calculation, and appropriate data reduction:

- Verify that all readings/outputs are accurate.

- Ensure proper error correction or data change (i.e., one line through, dated, initialed, and explained as appropriate).

- Select appropriate formulas for calculating final results, correct for background and/or interference (e.g., compton effects for gamma energy analysis and inter-element correction for inductively coupled plasma), and document calculations and results. These formulas shall be included as an integral part of each method standard operating procedure.

- Verify that data are accurately transcribed into notebooks, forms/benchsheets, or spreadsheets.

8.2.1   Significant Figures

Significant figures reflect the limits of the particular analysis method. Basic rules for significant figures and for calculating values and retaining the number of significant figures are provided in *Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications* (ASTM 1993). Reported values should contain only significant figures. The

number of significant figures should not exceed data needs as specified in project DQOs, SAPs, or other planning documents.

Recognizing that vendor-supplied software may not meet the general rules for significant figures, the laboratory should work with the client to determine the best way to report results based on the project needs.

8.2.2   Rounding-Off Methods

When a figure is to be rounded to fewer digits than the total number available, the rounding-off procedure described in ASTM (1993) should be followed. If a different rounding method is used it shall be noted in the narrative. A brief description of the procedure follows.

- When the first digit discarded is less than five, the last digit retained should not be changed.

- When the first digit discarded is greater than five, the last figure retained should be increased by one.

When the first digit discarded is exactly five, followed only by zeros, the last digit retained should be rounded upward if it is an odd number, but no adjustment made if it is an even number.

8.2.3   Data Review

Data review refers to the process of determining whether data conform to requirements specified in the project DQOs, SAPs, or other planning documents. All individuals involved in generating the data are responsible for the quality of the work they contribute to the final report.   Initial data review is conducted by the analyst during data collection. Individuals involved in steps such as data entry, data transcription, reduction, and project file assembly should review their work product before they forward it to the next stage in the reporting process. If they have questions about the work product from preceding steps in the process, they should undertake to resolve them before forwarding their work product to the next stage.

All data reported to the client shall be technically reviewed by someone other then the individual(s) who generated the results. Analysis performed in a field or fixed laboratory should receive independent review prior to being released to the project staff. In applications where data is being used in real-time to support project activities (most often *in situ analysis* supporting site remediation, screening for hot spots, etc.), procedures shall be in place that describe the types of real-time calibration confirmation and/or QC performance that the field analyst must have documented before providing data directly to project personnel in real-time. If the data generation is sufficiently complex or the intended use is critical to the project (pass/fail regulatory criteria, health and safety, etc.) it is recommended that a second, qualified individual be available to conduct an independent review within the time frame needed by the project.

The requirement for independent data review by the field analytical organization is not suspended in cases where real-time reporting is used. The data shall be reviewed by the appropriate analytical staff after it has been reported to the project staff. This review shall be expedited to ensure that important project decisions are not based on analytical data that are not correct. For example, if the standards used to confirm calibration of a measurement process are discovered to be incorrect or that a miscalculation occurred in converting raw data to reportable results, the mistake must be corrected immediately or risk continued incorrect real-time data reporting and project decision making. Where possible, such review should occur no later than the end of the working day, but definitely before the beginning of the next working day.

All review and reporting procedures shall be described in project documents, field, or laboratory organization standard operating procedures. In the event data needs to be reported without the prescribed review (e.g., emergency turnaround, inability to conducted timely review of real-time data within 24 hours of reporting) the data shall be marked as "Draft" or otherwise noted to indicate that the necessary reviews have not been, or will not be, completed according to procedure.   Errors discovered in real-time data that have been reported to the client shall be reported to the client immediately upon discovery with an estimate of the type of error that occurred. The errors shall then be referred to the analyst or other appropriate individual for corrective action (see Volume 1, Section 5.0). Timely notification of project personnel is critical to prevent potentially hazardous or regulatory non-compliant decisions to be made while the field analytical organization carries out the necessary corrective action.

The data review process shall incorporate the following elements.

- Data shall be reviewed according to the analytical organization's approved procedures to verify that calculations are correct and to detect transcription errors.

- Data shall be reviewed against applicable criteria for calibration, continuing calibration verification, QC, and other method criteria as appropriate to verify that the preparative and/or analytical system is performing acceptably (see Section 7.0 of this volume for details). If QC samples do not meet established criteria, data within the batch shall be evaluated to determine if there were any adverse effects with respect to the client's requirements. The sample(s) shall be re-prepared and/or re-run or the data shall be reported with an explanation and appropriate qualification(s), which will be detailed in the narrative.

- Random checks shall be performed to verify calibration, data entry, calculations, and QC criteria.

8.3   DATA REPORTING

The analytical information reported shall include the measured parameters, the details of analysis, and the reported data values.

Inorganic results shall be reported as numeric values with appropriate data qualifiers if above the IDL. If the value is less than IDL, it can be reported as undetectable.

Radiochemical results shall be reported based on calculated concentration or activity values (whether negative, positive, or zero) using the appropriate blank for each nuclide. The measured activity or concentration should be reported with estimates of total propagated uncertainty, but without comparison to the estimated a priori MDC. The MDC should not be reported to the client in lieu of low-level measurements.

8.3.1   Data Reporting Documentation

The reporting requirements for analysis shall be identified in the standard operating procedures describing the method or in project DQOs, SAPs, or other planning documents. These requirements may be significantly abbreviated from the requirements normally associated with fixed laboratory analysis. This is because the contents of the reports (and the supporting project documentation not included in the report to the client) should be agreed upon in advance by the project team and the field analytical organization. The contents of the reports plus the unreported information retained as part of the permanent record must demonstrate that the data meets the quality requirements specified in the planning documents.

The reporting requirements for data generated to support regulatory or compliance needs are generally more extensive than those associated with site screening or remediation support activities. This is characteristic of the graded approach to QA/QC embodied in the use of the DQO process. The list of report requirements for the two types of data (real-time decision making and regulatory or compliance support) are presented as a two-stage graded approach. In the first stage, the report requirements normally needed for real-time decision making are listed. The second stage material is the additional information normally required for submission in support of regulatory-based activities. Project-specific needs may be added to either stage, but the information identified in the following list is the minimum that is required by DOE to support the Hanford clean-up mission.

8.3.1.1   Report Requirements for Real-Time Project Decision Making. Project DQOs frequently identify activities that are driven by real-time data acquisition, reporting, and decision making. Examples of this are field or laboratory screening activities to identify potential hot spots for more detailed sampling and analysis and release of waste materials for disposition during remediation activities. These types of data needs are normally performed at Hanford using *in situ analysis* and/or *field analysis* in a field laboratory. The minimum report requirements for this type of data are:

- Laboratory identification. For analytical laboratories or groups providing *in situ* analytical services, the group should be identified by name (i.e., radiological counting facility) or project name (project XYZ field analysis group) and the name of the company providing the service (Waste Management Hanford, BHI, etc.).

- Name, title, and address of recipients of the report.

- Project name.

- Unique sample identifier, such as map grid coordinates or descriptive sample name, traceable to a site map (groundwater monitoring well #1, GW1, SE quadrant abandoned concrete pad #ABC). This applies to *in situ analysis* or *field analysis* where the field analyst performs the *in situ analysis* or collects the sample(s) and later performs the *field analysis* without the sample leaving their possession. See the additional requirements listed below for samples collected by a third party and relinquished to the analyst in the field or fixed laboratory.

- Analytical results and date of analysis (for each sample) with units (reported with an appropriate number of significant figures), and associated uncertainty where appropriate.

- Detection limits or screening levels, as appropriate.

- Method references.

- Statement the all calibration and QC criteria were met for the reported results.

- Signature and title of person accepting responsibility for the report contents.

- Date of issue.

8.3.1.2   Report Requirements for Regulatory or Compliance Activities. In addition to the ten information requirements listed in the preceding section, the following items of information are required to support activities associated with regulatory drivers such as waste characterization, waste shipment, permits, and site closure. These are:

- Chain-of-Custody form, including unique site identification, name, date and time of sample collection, unique client sample number, name of sampler(s), and names and organizations of people who accepted custody of the sample prior to arrival at the laboratory. Note: This information may be documented by providing a copy of the COC completed in the field.

- Sample information including unique laboratory identifier cross-referenced to client identification number, and date of sample receipt. Note: This information may be provided by providing a copy of the COC and associated sample receipt records after completion by the sample receiver.

- Date(s) of sample preparation.

- Identification of any amended test results.

- Identification of subcontracted results if applicable.

- Appropriate QC results (correlation with sample batch shall be traceable and documented).

- Appropriate data qualifiers with definitions and a narrative on the quality of the results if applicable.

- Additional data reporting, (i.e., the percent moisture/solid or correction for equivalent dry weight) as appropriate.

8.3.2   Emergency Reporting

An immediate data reporting system shall be established between the laboratory and the client to address emergency turnaround situations. The type of information, level of approval, data reporting format, and means of delivery shall be discussed and agreed upon between the laboratory and the client.

8.4   COMMON DATA QUALITY CALCULATIONS

This section provides various formulas that are typically employed to compute QC parameters that are used to assess data quality. These quality control parameters should be monitored, evaluated, and/or trended on a short-term and long-term bases. For example, system contamination control (blank or background activity), precision, accuracy, spike recovery, tracer or carrier yield recovery could be evaluated based on method, matrix, and activity or concentration level. Such activities provide a basis for continuous quality improvement and insight on overall laboratory performance.

8.4.1   Precision

Precision has been defined in Section 1.0. Sample precision is estimated by using duplicates, matrix spike duplicates or replicates. Samples used to calculate precision should contain concentrations of the analytes of interest above the MDC or EQL. The precision of a method in a given matrix is expressed as the relative standard deviation (RSD) or the relative percent difference (RPD).

In addition to precision determined by the sample duplicate or matrix spike duplicate, precision for the standards (e.g. laboratory control sample, continuing calibration verification standard) can be calculated and used to monitor quality control of the analytical measurement system over

time. Precision of the sample can also be monitored for long-term quality control, but should be based on method, matrix, and activity/concentration in the sample.

8.4.1.1 Relative Standard Deviation

The RSD is used when at least three replicate measurements are performed on a given technique. The RSD is computed using the following equation:

$$RSD = \frac{s}{\bar{x}} * 100$$

where

s	=	Standard deviation with n - 1 degrees of freedom
n	=	Total number of observed values
	=	Mean of observed values.

8.4.1.2 Relative Percent Difference

The RPD is used when two measurements exist. The RPD is generally used to express the precision of duplicate or spike duplicate samples. The RPD is computed using the following equation:

$$RPD = \frac{\left| x_1 - x_2 \right|}{\bar{x}} * 100$$

where

$x_{1,2}$	=	Observed values
	=	Mean of observed values.

8.4.2 Accuracy

8.4.2.1 Method Accuracy Based on Sample Spike

Accuracy has been defined in Section 1.0. Accuracy for the sample is expressed as the percent recovery (%R) of a matrix spike (or matrix spike duplicate) sample. The percent recovery is calculated based on the following equation:

$$\% \, R = \frac{(SSR - SR)}{SA} * 100$$

where

SSR   =   Spiked sample result
SR   =   Sample result
SA   =   Spike added

8.4.2.2 Method Accuracy Based on Standard

The accuracy of an analytical method is expressed as the percent recovery of a standard (%R). The percent recovery of a standard is calculated according to the following equation:

$$\% \, R = \frac{A_m}{A_k} * 100$$

where

A_m =   Measured value of the standard analyte
A_k =   Known value of the standard analyte.

Method accuracy obtained from either a sample spike or from a standard can be used to monitor quality control of the analytical measurement system over time. Sample accuracy should be tracked based on the method, matrix, and activity/concentration when it is used for long-term quality control monitoring.

8.4.3 Yield Recovery (Radiochemistry only)

Yield percent recovery (%Y) of a tracer or carrier in radiochemical analysis is a measure of the effectiveness of separation methods for some radionuclides. It is expressed as the percent recovery and is generally used to correct the analyte recovery in the sample for radiochemical analysis. Yield percent recovery is calculated according to the following equation:

$$\% \, Y = \frac{T_m}{T_k} * 100$$

where

T_m =   Measured value of the tracer or carrier
T_k =   Known value of the tracer or carrier.

Yield percent recovery should be evaluated per procedure to monitor the effectiveness of the radionuclide separation. If tracer or carrier is not used on every sample, a historic yield percent recovery should be used as the correction factor for the sample analyte.

8.4.4   Measures of Agreement

8.4.4.1 Percent Difference

The percent difference (%D) is often used to compare one reference point to another (e.g., average RF from initial calibration compared to RF from continuing calibration listed in Section 7.1.2). The percent difference is calculated using the following equation:

$$\% \, D = \frac{|I - C|}{I} * 100$$

where

$$
\begin{aligned}
I \quad &= \quad \text{Observed value used as the reference point} \\
C \quad &= \quad \text{Compared value.}
\end{aligned}
$$

8.4.4.2 Bias

Bias (B) is often used to measure the deviation of a measured value from a known value or accepted reference value. Bias can be assessed by comparing a measured value to an accepted reference value in a sample of known concentration or by determining the recovery of a known amount of analyte of interest spiked into a sample. Thus, the bias caused by the matrix effects based on a matrix spike is calculated using the following equation:

$$B = (X_s - X_u) - K$$

where

$$
\begin{aligned}
X_s \quad &= \quad \text{Measured value (e.g., spiked sample)} \\
X_u \quad &= \quad \text{Miscellaneous contribution (e.g., sample contribution)} \\
K \quad &= \quad \text{Known value (e.g., true spiked value).}
\end{aligned}
$$

If no miscellaneous contributions exist, X_u would be zero.

8.4.4.3 Mean Difference

Mean difference may be used to compare two duplicate results and is generally used for radiochemical analysis. The mean difference takes into account the uncertainty associated with each measurement. The mean difference is compared based on a two-sided z-test for a known population (Steel and Torrie 1960) and is calculated using the following equation:

$$MD = \frac{\left| R_1 - R_2 \right|}{\sqrt{(a_1^2 + a_2^2)}}$$

where

R_1	=	First sample result
R_2	=	Second sample result
a_1	=	One sigma uncertainty of first result
a_2	=	One sigma uncertainty of second result.

If the MD is greater than or equal to 1.96, a 95% confidence exists that the two results are not equal.

8.4.5   Detection Limit Considerations

Detection limit determinations are performed to give the laboratory, and subsequently the data user, information regarding the reliability of low-level results reported. A variety of approaches may be used, each of which portrays method sensitivity at low concentrations differently. This section describes several typical detection limit determinations. Each laboratory shall document which approach it employs and describe how the determination is applied (i.e., performed in sample matrix or performed using low-level standards).

8.4.5.1 Inorganic and Organic Methods

8.4.5.1.1 Method Detection Limit. The MDL is defined as "the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is greater than zero" (SW-846, consistent with the requirements specified in 40 CFR 40 Appendix B to 40 CFR 136) and is briefly described in the following text.

The concentration of the MDL for the analyte of concern can be estimated by using one of the following:

An instrument signal-to-noise ratio within the range of 2.5 to 5

The region of the standard curve where there is a significant change in sensitivity (i.e., a break in the slope of the standard curve).

When determining the MDL, a minimum of three analyses are required in a matrix spiking with the analyte of interest at a concentration three to five times the estimated MDL. Whenever possible, the matrix should be the same as or very similar to the sample matrix. All sample processing steps of the analytical method shall be included in the final determination of the MDL.

Variance (S^2) is determined from the replicate measurements, as shown:

$$S^2 = \frac{1}{(n-1)} \left[\sum_{i=1}^{n} (X_i - \overline{X})^2 \right]$$

where

    X_i =   With measurement of the variable X
            =   Mean of observed variable X.

The MDL should be determined by the following equation:

$$MDL = t_{(n-1, a=.99)} * (s)$$

where

    $t_{(n-1, \alpha=.99)}$ =   One-sided t-statistical value appropriate for the number of samples used to determine standard deviation

    s   =   Standard deviation obtained from the MDL replicate measurements.

Each laboratory shall document its approach when employing the MDL determination. The term MDL shall only be employed when all method steps (sample preparation through analysis) are tested. The laboratory shall also document whether the determination was performed using client samples or standards. Results falling below the MDL should be reported as not detected.

8.4.5.1.2 Instrument Detection Limit. IDL is determined by spiking reagent water with each analyte of concern. The following considerations apply to the selection of the IDL standard:

Concentration of the IDL standard should be at least equal to or in the same concentration range as the estimated IDL

Concentration of the IDL standard should be in the region of the standard curve where there is significant change in sensitivity.

A minimum of seven aliquots of the IDL standard are required to determine the IDL. The IDL standards are run through the analytical process only. The IDL is calculated the same as the MDL. In cases such as some organic analysis and mercury and cyanide determinations, the IDL standard should be subjected to preliminary extraction, digestion, and/or distillation. Each laboratory shall document whether the IDL includes the entire method (i.e., sample preparation through analysis) or only the analytical process. Results falling below the IDL should be reported as not detected.

8.4.5.1.3 Estimated Quantitation Limit. The estimated quantitation limit (EQL) is the lowest concentration of an analyte that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The analyte concentration at the estimated quantitation limit is determined using the following guidance: 1) 5 to 10 X MDL or IDL, or 2) the lowest non-zero standard in the calibration curve.

Each laboratory shall document the approach used to determine the EQL. EQLs reported with client data shall reflect all method dilution factors (i.e., dilution factors resulting from sample preparation).

Results falling between the IDL (or MDL) and the EQL should be reported with appropriate qualification (e.g., flag, footnote).

8.4.5.2 Radiochemistry Methods

8.4.5.2.1 Decision Level Count Rate. The decision level count rate (DLR) is defined as a 95% confidence limit for a critical decision level. This level is used for making a decision as to whether a sample emits radiation above the appropriate blank background level. The decision should be based solely upon whether the net count rate observed for that sample exceeds this DLR. The DLR is calculated as shown below:

$$DLR = 1.65 * \sqrt{\frac{R_b}{T_b} + \frac{R_s}{T}}$$

where

R_b =   Background count rate
R_s =   Sample count rate
T =   Sample count time
T_b =   Background count time.

When counting a sample containing no analyte (radionuclide) of interest, R_s is assumed to be equal to R_b. The DLR can be simplified as shown below:

$$DLR = 1.65 * (S_b) * \sqrt{2}$$

where

S_b =   Standard deviation of background (or appropriate blank) count rate for the counting time (T).

For the purpose of interpreting whether an individual sample measurement is different from its appropriate blank, it is recommended that the laboratory compare the net sample count rate with a decision level count rate calculated using the sample specific "appropriate" blank. The "appropriate" blank should include measurement interferences from impurities (e.g., elevated compton continuum, channel crosstalk from higher energy alpha particles measured by liquid scintillation) that are not typically known *a priori* or included in the nominal *a priori* DLR limit. This "true" decision level for the sample is different from the nominal *a priori* decision limit. For some measurement processes, the determination of the "true" appropriate blank for each sample may be impractical. However, every effort should be taken to properly assess the parameters of the appropriate blank.

8.4.5.2.2 Minimum Detectable Activity. The minimum detectable activity (MDA) has been defined as a level of activity that is practically achievable by a measurement system. The sample MDA generally is applied as the mean (expected) activity of samples having a 5% probability of escaping detection and 5% probability of false detection. The MDA is calculated based on Currie's (1968) formula and is simplified to the following two equations when the counting time in the sample is the same as in the background.

$$MDA = [(\frac{2.71}{T}) + (2 * DLR)] / K$$

or

$$MDA = [(\frac{2.71}{T}) + (4.65 * S_b)] / K$$

where

T   =   Sample count time
K   =   Detector calibration factor (e.g., count rate/disintegration rate)
S_b =   Standard deviation of background count rate for the counting time (T).

When T_b is not equal to T_t, MDA is calculated as shown below.

$$MDA = \underline{2.71 + 3.3} \frac{\sqrt{(R_b * T_b) * \left(1 + \dfrac{T_b}{T_t}\right)}}{\boldsymbol{x} * b * T_t * k}$$

where

R_b	=	Background count rate
T_b	=	Background count time
T_t	=	Sample count time
ξ	=	Counting efficiency
b	=	Abundance
k	=	conversion factor to convert to desired units.

The minimum detectable concentration (MDC) is defined as the mean concentration of samples having a 5% probability of escaping detection and 5% probability of false detection.

$$MDC = \frac{MDA}{q * Y * decay}$$

where

q	=	Sample quantity (e.g., g or mL)
Y	=	Chemical yield
decay	=	Decay factor (correction for radioactive decay to reference date).

Software provided by vendors may have variations of the above formula. A vendor- provided software or data reduction package is adequate for data calculation.

8.4.5.2.3 *A Priori* and *A Posteriori* Concepts and Information. Decision level count rate, MDA, and MDC are considered as *a priori* (before the measurement). The estimation of these quantities requires specification of nominal values of a number of parameters (e.g., background count rate, count time, estimated interferences, chemical recoveries, decay times). The true appropriate blank for a measurement process includes estimates of the nominal levels of any interferences that may be present in a sample batch.In a number of situations, regulatory limits or contract specifications may require that the measurement process meet or exceed certain MDC limits for the sample batch of interest. Because these determinations require that some preliminary measurements be made, one finds that the assessment of *a priori* detection limit parameters for future measurements may require the knowledge of *a posteriori* information concerning the nominal characteristics of the sample batch gained from preliminary measurements.

The question of whether the sample contains net activity is best answered by comparing the measurement result to the decision level or considering the confidence interval for the measurement result, not by comparing the result to the estimated MDA or MDC.

8.4.5.3 Limit of Detection

In some cases, the limit of detection is used. The limit of detection is defined as an analyte signal that is three times the standard deviation of its measurements above the corresponding well-characterized blank response (Keith 1991).

The limit of detection represents a criterion for detection decision, i.e., deciding whether to classify a result as detected or not detected when the observed signal is close to that obtained for blank measurements (i.e., similar to background noise).

8.4.6   Uncertainty

Uncertainty is expressed as the range of values in which the true value is estimated to lie. The uncertainty estimate consists of two components, systematic and random variability. Each contributing source of uncertainty is expected to be distributed over its range. Each systematic component can be estimated in terms of the measurement result for the contributing source of uncertainty. The analytical systematic component can be estimated using standard or spike recovery. The random analytical component can be estimated from replicate measurements of a sample. The total uncertainty is calculated as the square root of the sum of the squares of random and systematic variabilities as shown in the following equation. The component of uncertainty has to be expressed in the same unit designation (e.g., concentration percentage).

$$\text{Total uncertaint } y = \sqrt{(s_x^2) + \sum_{j=1}^{q} \boldsymbol{d}_j^2}$$

where

s_x = Standard error
q = Number of systematic uncertainty component
δ = Systematic uncertainties.

Uncertainty is commonly used in the radiochemical analyses to express method and counting error. The total random uncertainty is obtained by propagating the individual variance (s_i^2) and is expressed as the standard error based on multiple determinations of x. However, the typical radiochemical methods used are not sufficient to separate systematic and random uncertainties such that biases can be corrected. Uncertainty will be measured, or uncertainty will be estimated if it cannot be measured.

8.4.7   Control Charts

Control charts provide the analyst with early warning of impending problems in a preparative or analytical method. Each laboratory shall document its policy regarding the use of control charts. The laboratory's policy shall articulate the manner in which it will deal with statistical outliers. Blank spike/laboratory control sample performance for all routine preparations shall be monitored via control charts. Radiochemical laboratories shall also monitor calibration verification standards (i.e., counter control standard for radiochemistry). In those cases where the analytical technique involves a large number of analytes (e.g., ICP, GC/MS) the laboratory may select a subset representative of the total for control charting. Additional information on the application, development, and use of control charts can be found in the Ecology manual entitled, *Procedural Manual for the Environmental Laboratory Accreditation Program*. Tabulation of performance statistics can be used in lieu of a control chart.

Laboratories shall document and apply procedures for estimating uncertainty. The rigor associated with the protocol will depend upon the relative contribution from each source of error. When a laboratory implements an industry-recognized method which already specifies the limits for major sources of uncertainty then the laboratory would meet this requirement providing it reported results consistent with the method. In such cases the major sources of uncertainty would still be provided to the client (e.g., in terms of sample precision and accuracy results).

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9.0   DATA VERIFICATION, VALIDATION, AND ASSESSMENT

This section provides guidance to the field analytical organization in the areas of data verification, validation, and assessment. While the optimization bears no direct responsibility for validation or assessment, it plays an important supporting role.

Data verification is the process where the analytical service organization confirms that all work requested by the client has been performed properly. The SAP, QAPjP, client communications, chain-of-custody forms, and other appropriate documents establish the specific information and quality requirements for each project. Verification is performed against these requirements using the information contained in the analytical report(s) to the client and all supporting documentation generated and retained by the analytical organization. This material is the same as that used in the QA review process. Verification should not result in any quality findings that are not found during a thorough QA review. The analytical service organization should establish a procedure to conduct this verification.

Data validation is the process where the client or project submits the analytical data (including appropriate supporting documents provided by the laboratory) to a qualified, independent third party for review. The third party compares the information against the project DQO and other planning documents, the laboratory QA/QC requirements, and prevailing industry or regulatory standards. This comparison results in a validation report identifying any quality gaps between the client's expectations (DQOs and planning documents), laboratory QA/QC program, and industry standards and the analytical data and supporting information. The analytical service organization has no role in the validation process beyond the timely submission of copies of all information requested by the third party organization. The analytical service organization should be available to provide any additional information requested by the third party as the process proceeds. The service organization should establish a procedure to provide and track the information submitted to the client (or project) and the third party organization.

Data quality assessment (DQA) is the process where the client or project examines the analytical data, sampling data, and other appropriate information to determine if the data is adequate to its intended use. The DQA process evaluates that spatial and statistical distribution is evaluated and provides the basis for interpretation of the data. The analytical service organization should be prepared to provide any additional information requested by the client or project.

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10.0   REFERENCES

ASTM, 1993, *Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications*, ASTM E-29, American Society for Testing and Materials, Philadelphia, Pennsylvania.

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